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IRON ORES - DETERMINATION OF Fe-TOTAL BY NON MERCURY TITRIMETRIC METHODS

STATISTICAL EVALUATION OF INTERNATIONAL TEST DATA - 1982

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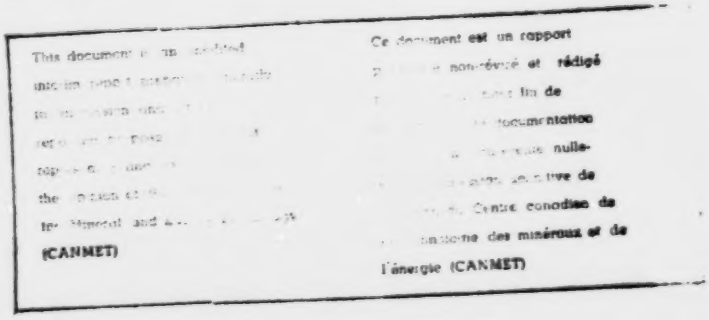
Analytical Methodology



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by

R. Sutarno* and W.S. Bowman**

SUMMARY

The precision and accuracies of two analytical methods for the determination of Fe-total in iron ores by non-mercury titrimetry using (a) Ag reduction and (b) $TiCl_3$ reduction have been estimated from the data resulting from international tests. These international tests involved 33 laboratories for the Ag-reduction method and 19 laboratories for the $TiCl_3$-reduction method and used a set of 5 iron ore samples containing 44 to 68% iron. It was found that these two methods have comparable accuracy with the following precision:

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	Ag reduction	$TiCl_3$ reduction
r(%Fe)	0.216	0.175
P(%Fe)	0.335	0.240
σ_r(%Fe)	0.076	0.062
σ_L(%Fe)	0.105	0.072

when r is the repeatability, P is the permissible tolerance, σ_r is the within-laboratory standard deviation and σ_L is the between-laboratory standard deviation.

INTRODUCTION

The ISO/TC 102/SC 2 (ISO Technical Committee on Iron Ores, Sub-committee 2 - Chemical Analysis) is currently developing standard methods of analysis for total iron content in iron ores by non-mercury titrimetric methods. Two candidate methods are being considered: (a) a method utilizing Ag reduction which is being developed by Working Group (WG)17B and (b) one using $TiCl_3$ reduction which is being developed by WG 16B.

It was decided at the tenth meeting of the sub-committee that, in order to compare the statistical parameters of these two methods, joint international tests would be conducted by these two WG's during the period of 1980-1982, using the same set of iron ore samples and the same experimental design (1). Thirty-three and nineteen laboratories participated in the international tests conducted by WG 17B and WG 16B, respectively. Each laboratory analysed two bottles of each test sample in duplicate. Five test samples with iron contents ranging from 44 to 67% were used.

PRINCIPLE OF THE ANALYTICAL METHODS

(a) Ag reduction (2)

The iron ore sample is fused with sodium peroxide in a zirconium crucible; the melt is leached with water. Addition of hydrochloric acid and warming results in complete dissolution. This solution is passed through a silver reductor. After addition of phosphoric acid - sulphuric acid mixture and diphenylamine sulphonate indicator, the total iron is determined by titration with a standard solution of potassium dichromate.

(b) $TiCl_3$ reduction (3)

Decomposition of the test portion:

a) Samples containing less than 0.05% vanadium: by sintering and fusion with alkali, leaching of the cold melt with water and dissolution in hydrochloric acid.

b) Samples containing more than 0.05% vanadium: by sintering and fusion with alkali, leaching of the cold melt with water, followed by filtration. Dissolution of the residue in hydrochloric acid.

Titration of iron:

Reduction of the major portion of the trivalent iron by stannous chloride and reduction of the remainder of the iron by titanium trichloride using indigo carmine indicator. Oxidation of the excess reductant with diluted potassium dichromate solution. Titration of the reduced iron with potassium dichromate solution using sodium diphenylamine sulphonate indicator.

STATISTICAL ANALYSIS

The analytical data collected by both WG's from their international tests during the period of 1980-1982 were statistically evaluated by the procedure described in document ISO/TC 102/SC 2 N 532E (4). This procedure is schematically illustrated in Figure 1. The raw data are also plotted in Figure 2. The boxed points in these figures signify that these data were suspected outliers and were not used to estimate the grand mean and precisions. The analytical results reported by WG 17B and for samples 79-3 and 76-17 show that these samples are inhomogeneous, i.e., that the between-

bottles variations for these samples are significant when estimated by the two-way ANOVA technique; as a consequence, a one-way ANOVA could not be performed with the result that some of the data were not used for the computation of the grand mean and precisions.

The results of this evaluation are summarized in the following tables:

- Tables 1a and 1b - list the grand means and precisions
- Tables 2a and 2b - list the suspected outliers
- Table 3 - lists the ratio between the grand means and precisions found by WG 17B to those found by WG 16B
- Table 4 - lists the comparison between the grand means and precisions estimated by the results of the current international tests with those obtained from the previous international tests
- Table 5 - lists the final estimates of precisions.

OBSERVATIONS

Based on the results of the present international tests the following observations are made:

(a) Accuracy of the method

Tables 1 and 3 show that the grand mean obtained by both methods are virtually identical with the exception of sample 79-3 where the grand mean obtained by Ag-reduction method is about 1% higher than that of $TiCl_3$ reduction. This difference is also statistically significant (Table 4).

(b) Repeatability

Both methods produce consistant within-laboratory precision. The repeatability, r, is virtually constant (Table 1 and Figure 3). The values of the r for the Ag reduction are greater than those of $TiCl_3$-reduction method for all samples (Table 3).

(c) Permissible tolerance, P

Results of both methods show that there are problems with samples 79-3 and 76-17. Ag-reduction results indicate that these samples are inhomogeneous. The between-bottles standard deviations were estimated to be 0.13 for sample 79-3 and 0.08 for sample 76-17. These are significant in relation to the within-laboratory standard deviation.

$TiCl_3$ reduction results did not indicate that these samples were inhomogeneous; however, they produced estimates for between-laboratories standard deviation (and thus the permissible tolerance) that are out of line (too high).

Figure 3 shows that when the results of sample 79-3 are included in the computation of the regression equation, both the between-laboratory standard deviation σ_L and the permissible tolerance, P have negative slopes. This is an uncommon occurrence in the functional relationship between precision and concentration level.

With the exception of sample 76-17, both P and σ_L for the Ag reduction are greater than those of the $TiCl_3$ reduction (Table 3).

(d) Outliers

The number of outliers indicated in Tables 2a and 2b are reasonable; they are within the limit of statistical error used in the test (5%).

(e) Comparison with previous results

It is unfortunate that the only common samples used for both the 1978-1980 and 1980-1982 international tests are identified as problem samples. Comparison of results for these samples (Table 4) should, therefore, be treated with caution. Table 4 shows that with the exception of the grand mean computed from the results of 79-3 by Ag-reduction - 1982 all other statistics are comparable. It implies that the problems encountered during the 1978-1980 tests (5,6) were still in existance during the 1980-1982 tests.

If one excludes the results of samples 79-3 and 76-17, the results of the 1980-1982 trials show that the precision of the Ag-reduction method is about the same as those of ISO 2597 (the current ISO standard method for Fe-total) which is based on experimental results reported in document 2 N 147E; and $TiCl_3$-reduction method shows slightly better precision (Table 5). The precision listed in Table 5 is the arithmetric average of the precisions derived from samples JSS 850-1, NBS 692 and SCH-1. Experimentally, therefore Table 5 is only valid for total iron contents ranging from 59-67%. Extension of this range will be based on chemical justification.

CONCLUSION

The results of the present joint international tests between WG 17B and WG 16B can be summarized as follows:

1) Both Ag-reduction and $TiCl_3$-reduction methods are caple of producing analytical results with equal accuracy.
2) Both methods seem to experience problems with samples 79-3 (0.9% Cu).
3) The $TiCl_3$-reduction method seems to have marginally better precision than the Ag-reduction method.

REFERENCES

1 - "Final report of the tenth meeting of ISO/TC 102/SC 2 - Iron ores/ chemical analysis, South African Bureau of Standards, Pretoria"; ISO/TC 102/SC 2 (Sect-214) N 655E; August 1981.

2 - "Iron ores - Redoximetry determination of total iron-mercury pollution free method"; ISO/TC 102/SC 2 (IWG 17-1) N 571E; April 24, 1979.

3 - "Iron ores - Determination of total iron content - Non-mercury titrimetric method"; ISO/TC 102/SC 2 (IWG 16-7) N 569E; March 2, 1979.

4 - "Procedure for statistical evaluation of analytical data resulting from international tests"; ISO/TC 102/SC 2 (WG 7-21) N 532E; August 1978.

5 - R. Sutarno and W.S. Bowman, "Iron ores - Redoximetry determination of total iron-mercury pollution free method - ISO/TC 102/SC 2 (IWG 17-1) N 571E - Statistical analysis of international test data"; Division Report MRP/MSL 80-81 (IR); CANMET, Energy, Mines and Resources Canada; June 1980.

6 - R. Sutarno and W.S. Bowman, "Iron ores - Determination of total iron - Non-mercury titrimetric method ISO/TC 102/SC 2 (IWG 16-7) N 569E, Statistical analysis of international test data"; Division Report MRP/MSL 80-123 (IR); CANMET, Energy, Mines and Resources Canada; September 1980.

7 - "Results of international tests on eight iron ore samples"; ISO/TC 102/SC 2 (Secretariat-42) N 147E; May 1968.

Table 1a

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E

SUMMARY OF PRECISIONS FOR ALL SAMPLES

SAMPLE	MEAN(X)	REPEATABILITY	PERMISSIBLE TOLERANCE	SIGMA-R	SIGMA-L
79-3	44.9191	.2081	.5139	.07354	.17398
76-17	67.0836	.2488	.3190	.08790	.09405
JSS 850-1	66.7805	.2214	.3303	.07823	.10277
NBS 692	59.6128	.2119	.2972	.07488	.09070
SCH-1	60.6477	.2149	.3784	.07592	.12245

THE REGRESSION EQUATIONS ARE:					CORR. COEFF.
R	=	.0012 X	+	.1484	.6721
P	=	-.0085 X	+	.8789	-.8849
SIGMA-R	=	.0004 X	+	.0525	.6717
SIGMA-L	=	-.0034 X	+	.3208	-.8956

Table 1b

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E

SUMMARY OF PRECISIONS FOR ALL SAMPLES

SAMPLE	MEAN(X)	REPEATABILITY	PERMISSIBLE TOLERANCE	SIGMA-R	SIGMA-L
79-3	44.4545	.1577	.4603	.05574	.15779
76-17	67.1076	.1739	.3722	.06144	.12412
JSS 850-1	66.7569	.1668	.1991	.05893	.05669
NBS 692	59.6058	.1635	.2052	.05779	.05991
SCH-1	60.6738	.1945	.3155	.06872	.10035

THE REGRESSION EQUATIONS ARE:					CORR. COEFF.
R	=	.0006 X	+	.1329	.4155
P	=	-.0077 X	+	.7694	-.6334
SIGMA-R	=	.0002 X	+	.0470	.4153
SIGMA-L	=	-.0030 X	+	.2771	-.6345

Table 2a - List of suspected outliers in the results of the international test on Fe-total by titrimetric method using Ag-reduction Document ISO/TC 102/SC 2 N 571E.

Sample No.	K_o	N_o	K	N	Outliers			σ_L/σ_r
					Laboratory		Individual result	
					Cochran (precision)	Dixon (accuracy)		
79-3*	33	132	27	108	-	-	-	2.4
76-13*	33	132	30	120	-	-	-	1.1
JSS 850-1	33	132	32	128	GER 1	-	-	1.3
NBS 692	33	132	32	126	-	SW 1	CND 2, SW 3	1.2
SCH-1	33	132	33	132	-	-	-	1.6

*Between bottles variation is statistically significant

K_o - No. of laboratories participated in the international test

N_o - Total number of observations submitted by the participating Laboratories for a particular test sample

K - No. of laboratory results used for ANOVA for a particular test sample

N - No. of analytical results used for ANOVA for a particular test sample

Table 2b - Summary of suspected outliers found in the results of the international test on Fe-total by titrimetric method using $TiCl_3$ reduction Document ISO/TC 102/SC 2 N 569E.

Sample No.	K_o	N_u	K	N	Outliers			c_L/σ_r
					Laboratory		Individual result	
					Cochran (precision)	Dixon (accuracy)		
79-3	19	76	17	68	AU 1, FR 3	-	-	2.8
76-17	19	76	18	72	-	GER 1	-	2.0
JSS 850-1	19	76	19	76	-	-	-	1.0
NBS 692	19	76	17	68	FR 5, GER 1	-	-	1.0
SCH-1	19	76	19	76	-	-	-	1.4

Table 3 - Ratios of the means and precisions resulting from international tests on Fe-total by titrimetric method using Ag reduction to those using $TiCl_3$ reduction*

Sample No.	Grand Mean $\bar{x}_{17}/\bar{x}_{16}$	Precision			
		r_{17}/r_{16}	P_{17}/P_{16}	$\sigma_{r_{17}}/\sigma_{r_{16}}$	$\sigma_{L_{17}}/\sigma_{L_{16}}$
79-3	1.01	1.32	1.11	1.32	1.10
76-17	1.00	1.43	0.86	1.43	0.76
JSS 850-1	1.00	1.33	1.66	1.33	1.81
NBS 692	1.00	1.29	1.45	1.29	1.52
SCH-1	1.00	1.10	1.20	1.10	1.22

*17 - Ag reduction
16 - $TiCl_3$ reduction

Table 4. Comparison between the grand means and precisions calculated from the results of international tests on Fe-total by titrimetric methods using Ag and $TiCl_3$ reduction conducted in the period from 1978-1980 and 1980-1982.

Method/ Year of international tests	Sample No.	Grand Mean 95% Confidence Interval			Precision			
		Low %	Mean $\bar{x}$ %	High %	r %	P %	σ_x^r %	σ_x^L %
Ag reduction -1980	79-3	44.288	44.361	44.433	0.254	0.416	0.092	0.135
Ag reduction -1982		44.840	44.919	44.998	0.208	0.514	0.074	0.174
$TiCl_3$ reduction -1980		44.358	44.438	44.518	0.190	0.491	0.068	0.170
$TiCl_3$ reduction -1982		44.372	44.454	44.537	0.158	0.460	0.056	0.158
Ag reduction -1980	76-17	66.957	67.095	67.232	0.217	0.753	0.078	0.266
Ag reduction -1982		67.039	67.084	67.128	0.249	0.319	0.088	0.094
$TiCl_3$ reduction -1980		67.016	67.072	67.129	0.212	0.345	0.077	0.112
$TiCl_3$ reduction -1982		67.044	67.108	67.171	0.174	0.172	0.061	0.124

Table 5 - Determination of Fe-total in iron ores by titrimetric methods described in documents ISO/TC 102/SC 2 N 571E, ISO/TC 102/SC 2 N 569E and ISO 2597 (7)
Precision estimated from international tests*

Method document	r %	P %	σ_r %	σ_L %
ISO/TC 102/SC 2 N 571 (Ag reduction)	0.216	0.335	0.076	0.105
ISO/TC 102/SC 2 N 569 ($TiCl_3$ reduction)	0.175	0.240	0.062	0.072
2 N 147E $SnCl_2$	0.232	0.506	0.084	0.172
2 N 147E H_2S	0.257	0.490	0.093	0.163

*Based on three samples with Fe-total contents ranging from 59 to 67%.

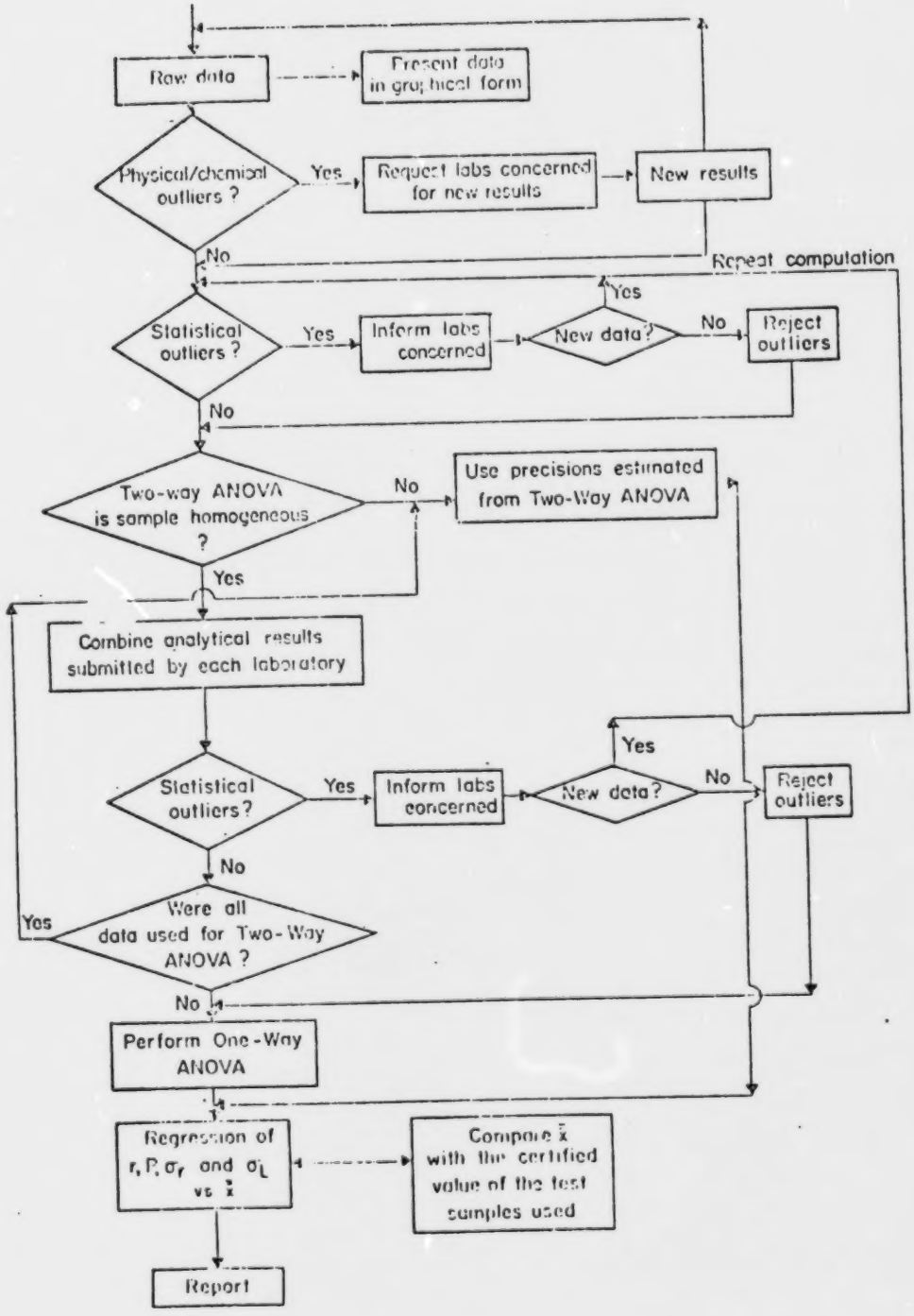
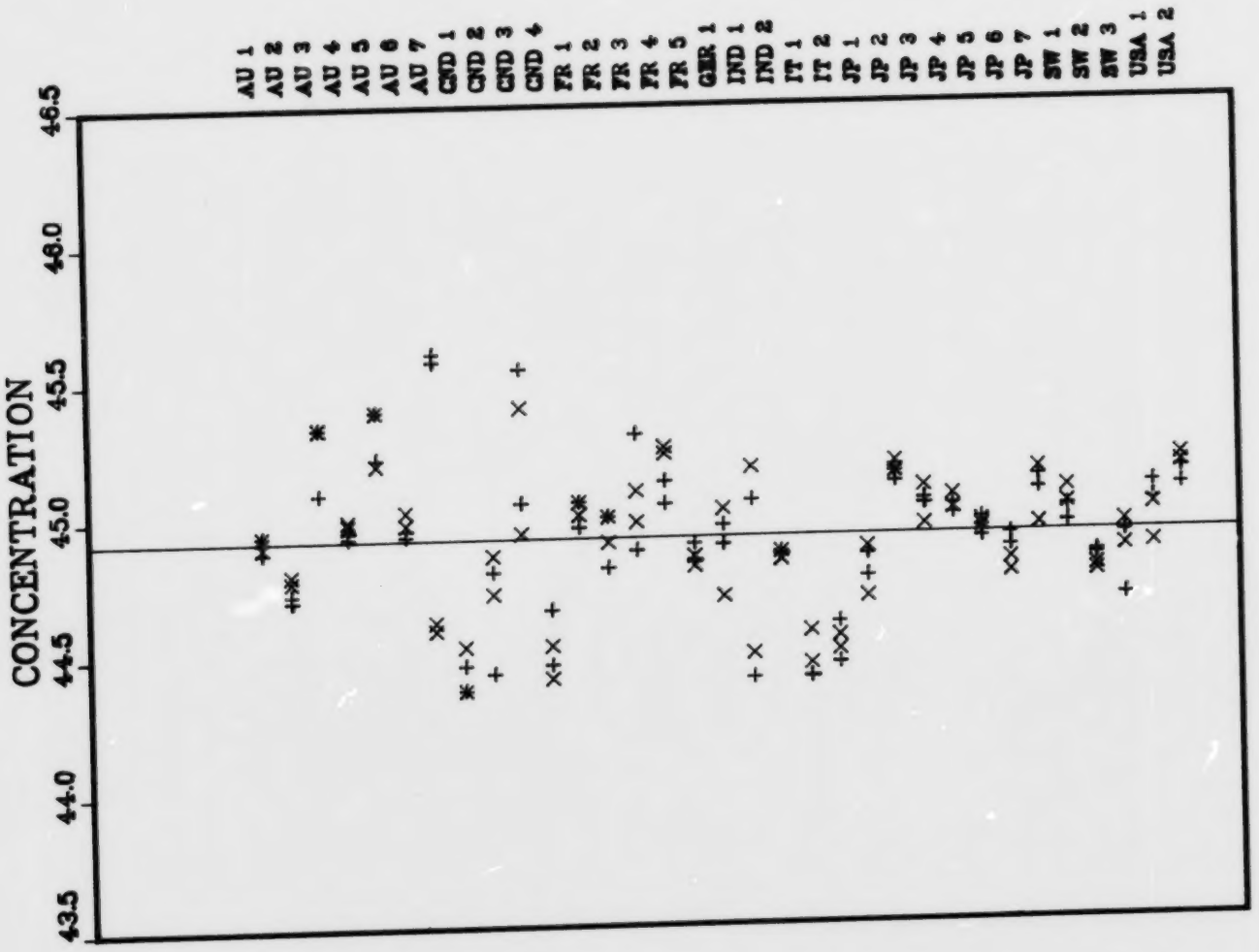


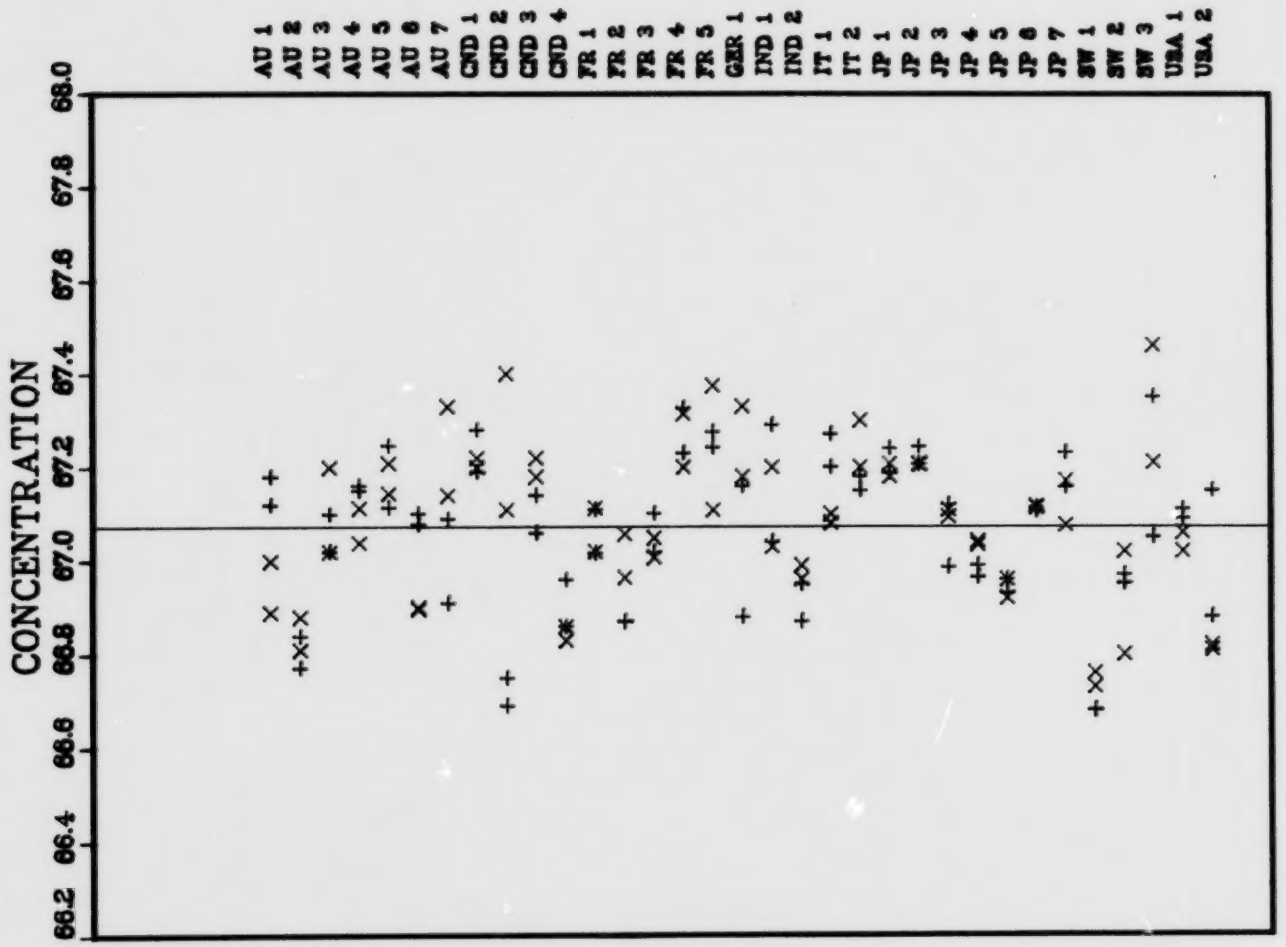
Figure 1. Flow chart of the procedure for statistical evaluation of analytical data resulting from international tests.



ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E

Figure 2.a.1

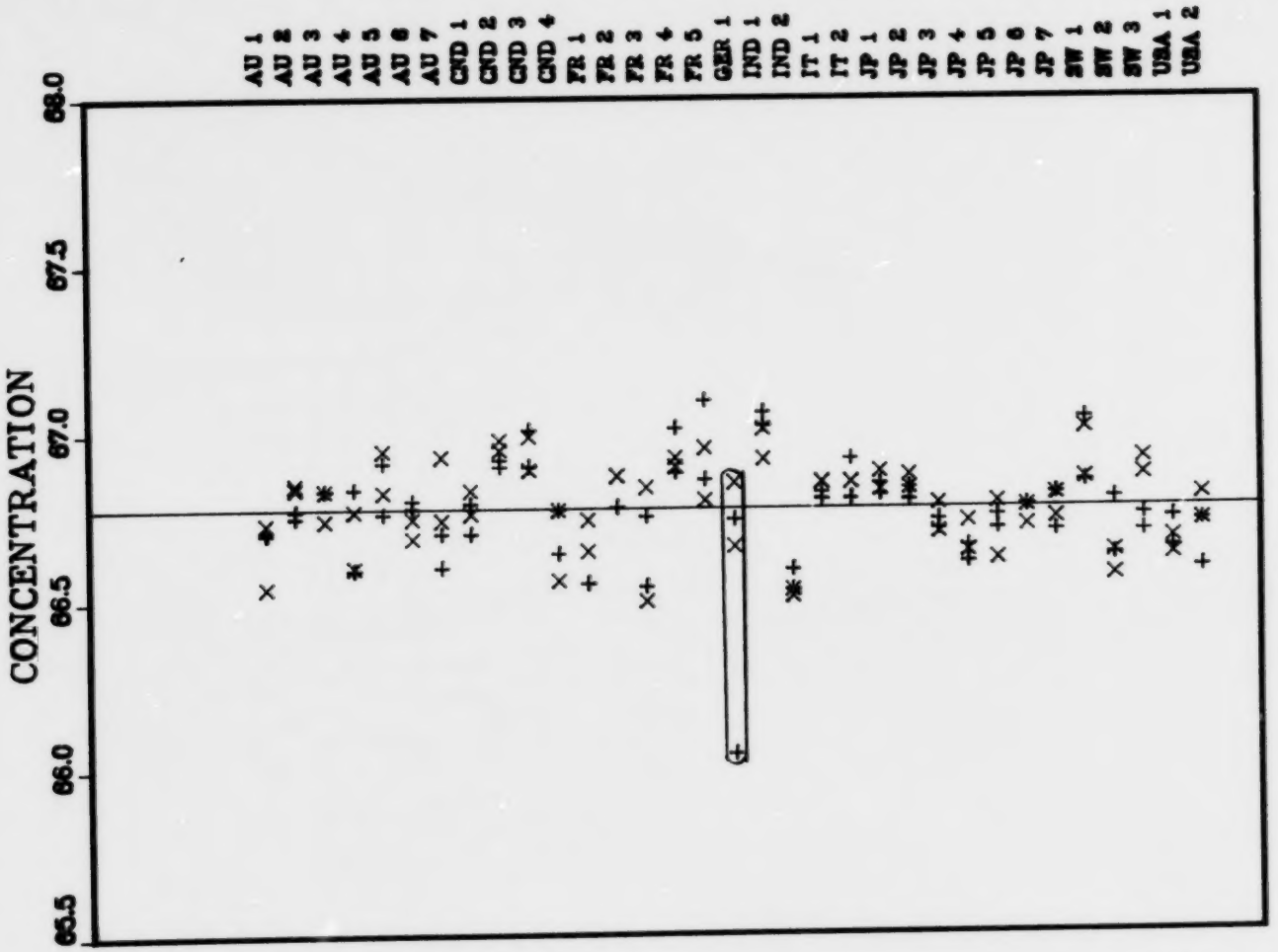
SAMPLE: 1. BACKGRUVAN 79-3



ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E

Figure 2.a.2

SAMPLE 2. SAVAGE JSS 852-1 76-17



ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E

Figure 2.a.3

SAMPLE: 3. MARCONA JSS 650-1

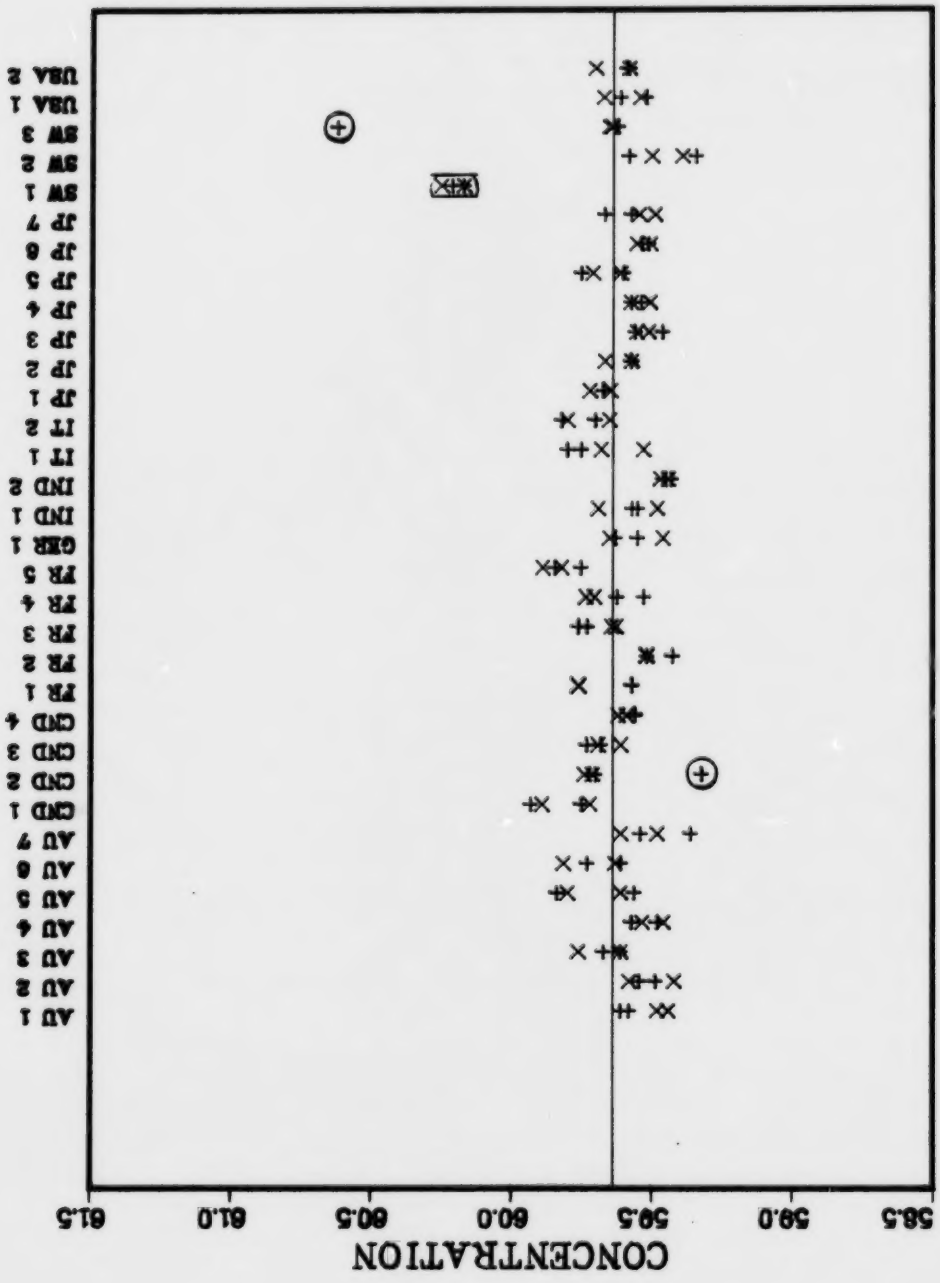

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692
CONCENTRATION
58.5
59.0
59.5
60.0
60.5
61.0
61.5
AU 1
AU 2
AU 3
AU 4
AU 5
AU 6
AU 7
CHD 1
CHD 2
CHD 3
CHD 4
FR 1
FR 2
FR 3
FR 4
FR 5
GER 1
IND 1
IND 2
IT 1
IT 2
JP 1
JP 2
JP 3
JP 4
JP 5
JP 6
JP 7
SW 1
SW 2
SW 3
USA 1
USA 2

Figure 2.a.4

ISO/TC102/SC2/WG1FB FE-TOTAL SILVER REDUCTION METHOD DOC ISO/TC102/SC2 N571E
SAMPLE: 5. CANADA SCE-1
CONCENTRATION
59.6 59.8 60.0 60.2 60.4 60.6 60.8 61.0 61.2 61.4 61.6
AU 1
AU 2
AU 3
AU 4
AU 5
AU 6
AU 7
CND 1
CND 2
CND 3
CND 4
FR 1
FR 2
FR 3
FR 4
FR 5
GER 1
IND 1
IND 2
IT 1
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Figure 2.a.5

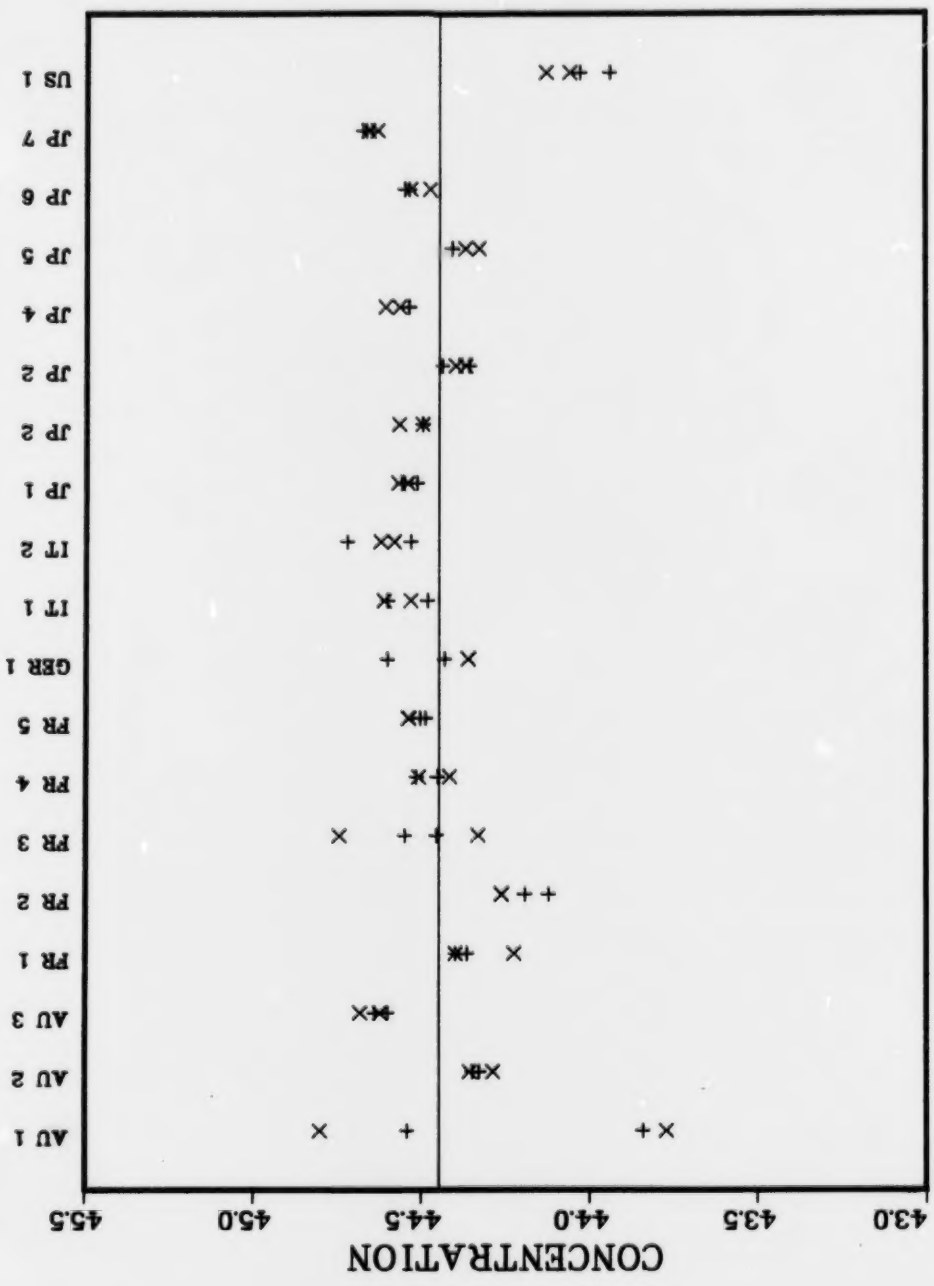

Figure :2.b.1

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E

SAMPLE: 1. BACKKGRUVAN 79-3

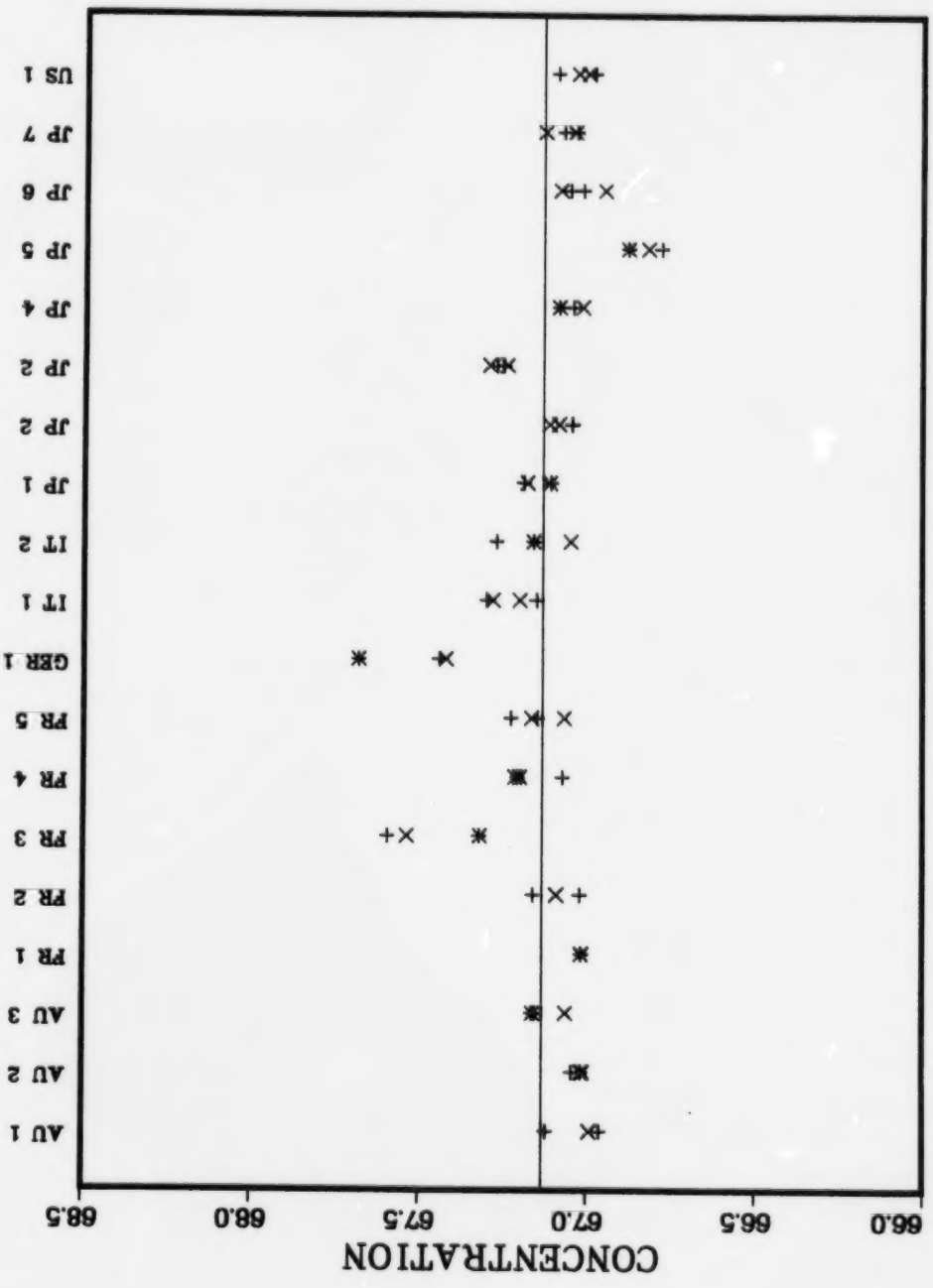

Figure 2.b.2

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N568E

SAMPLE: 2. SAVAGE JSS 852-1 76-17

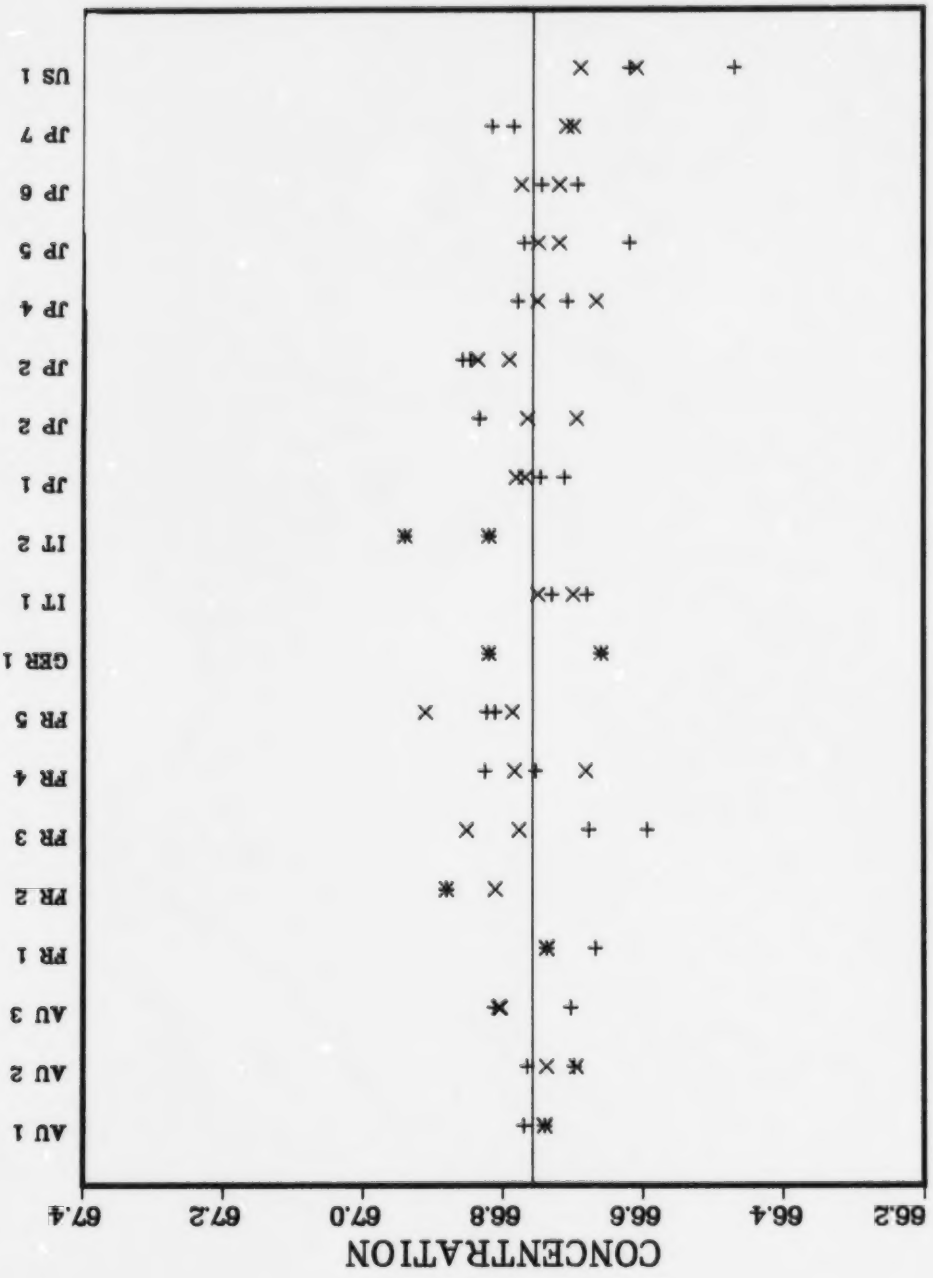

Figure 2.b.3
ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 3. MARCONA JSS 850-1

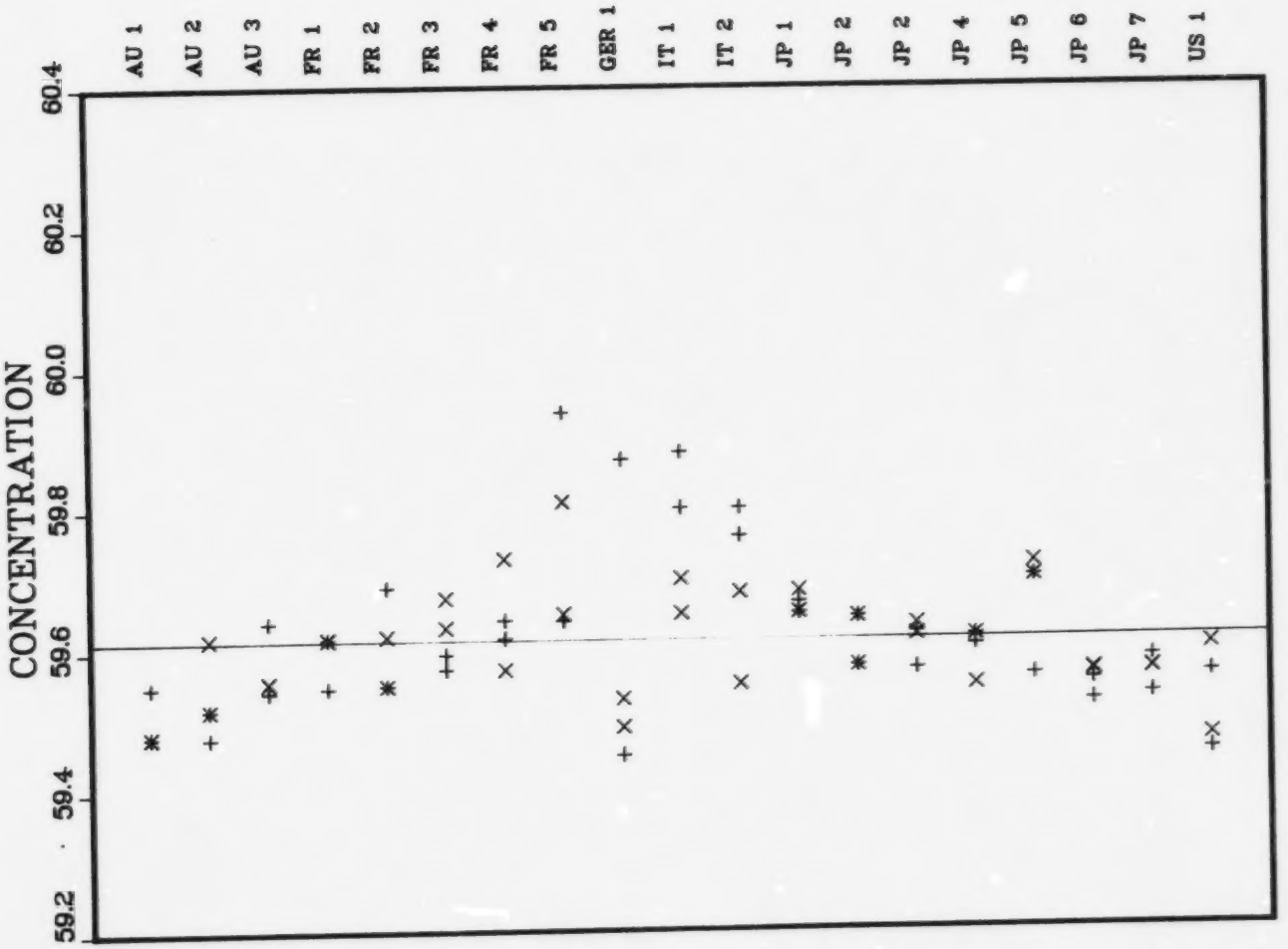

Figure 2.b.4

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E

SAMPLE: 4. LABRADOR NBS 692

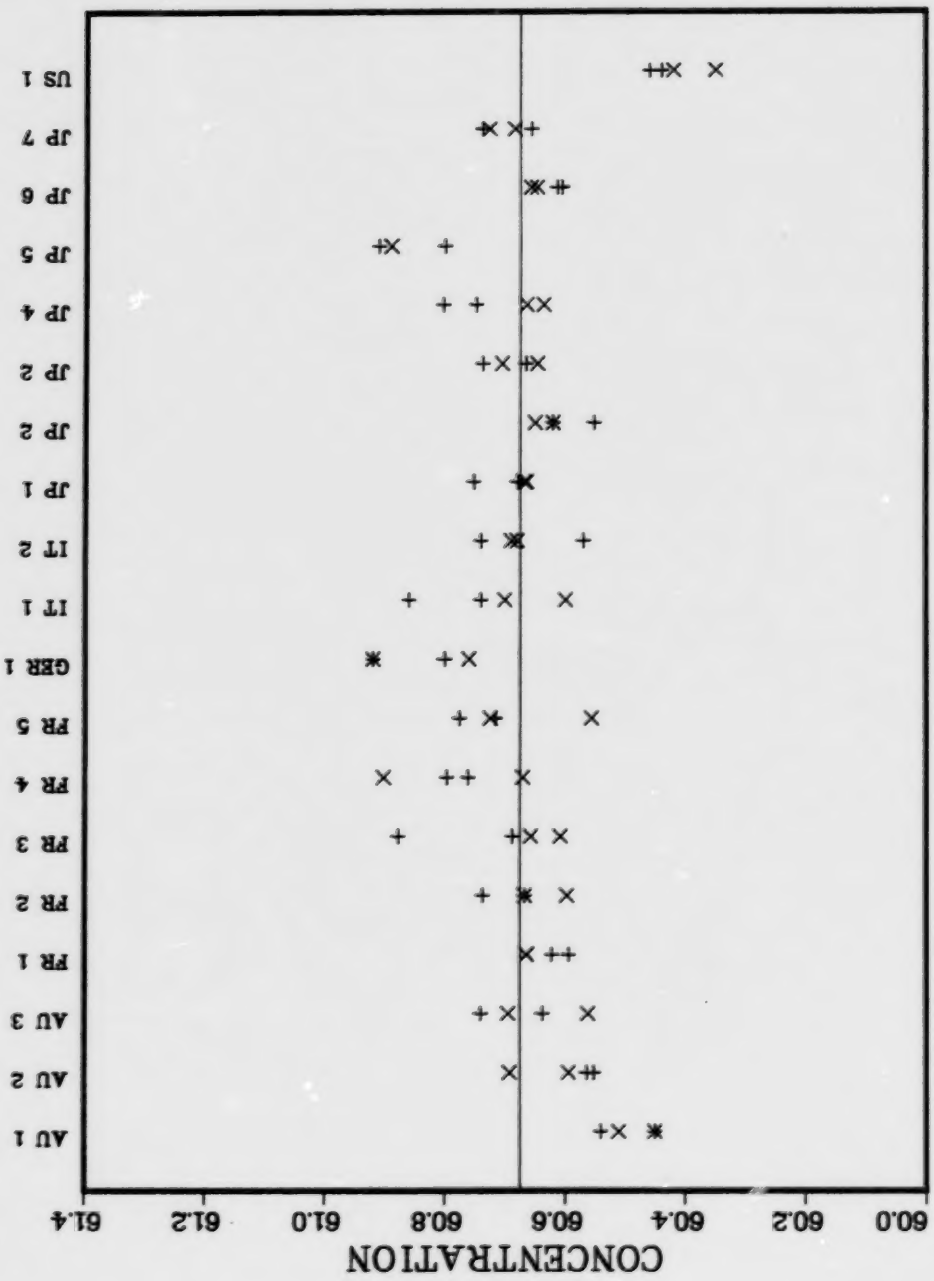

Figure 2.b.5

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E

SAMPLE: 5. CANADA SCH-1

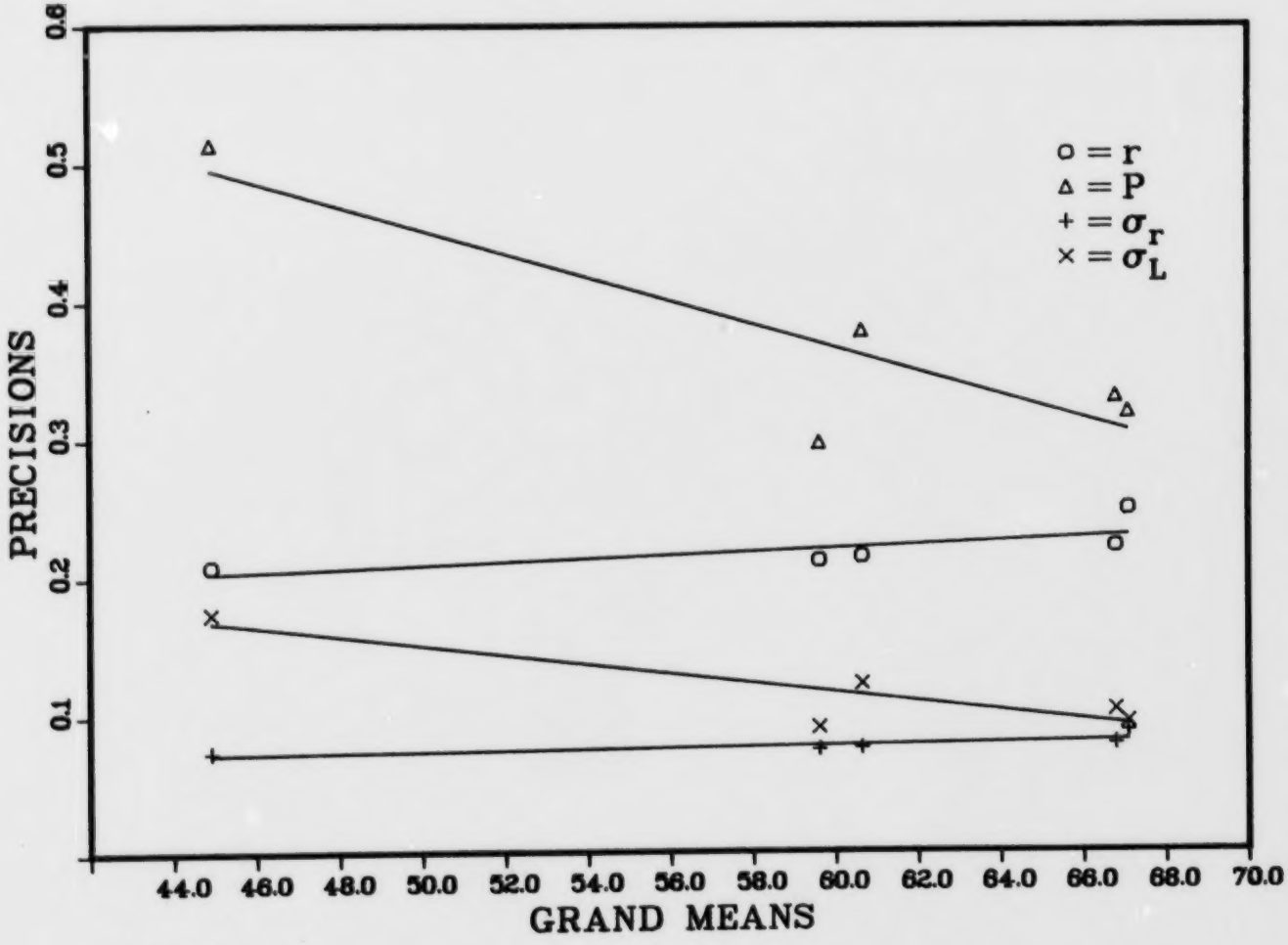

Figure 3.a.1

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E

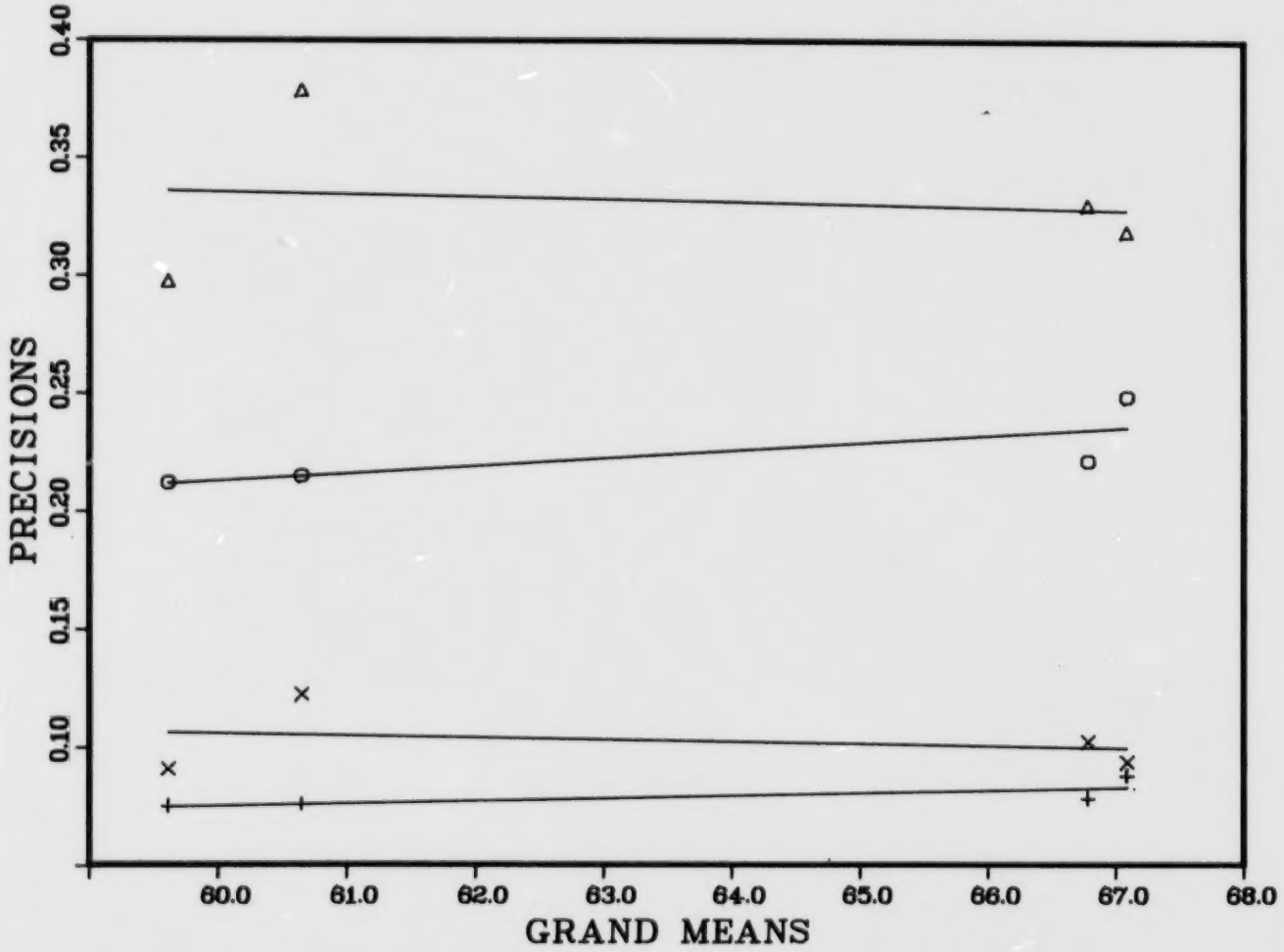

Figure 3.a.2

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E

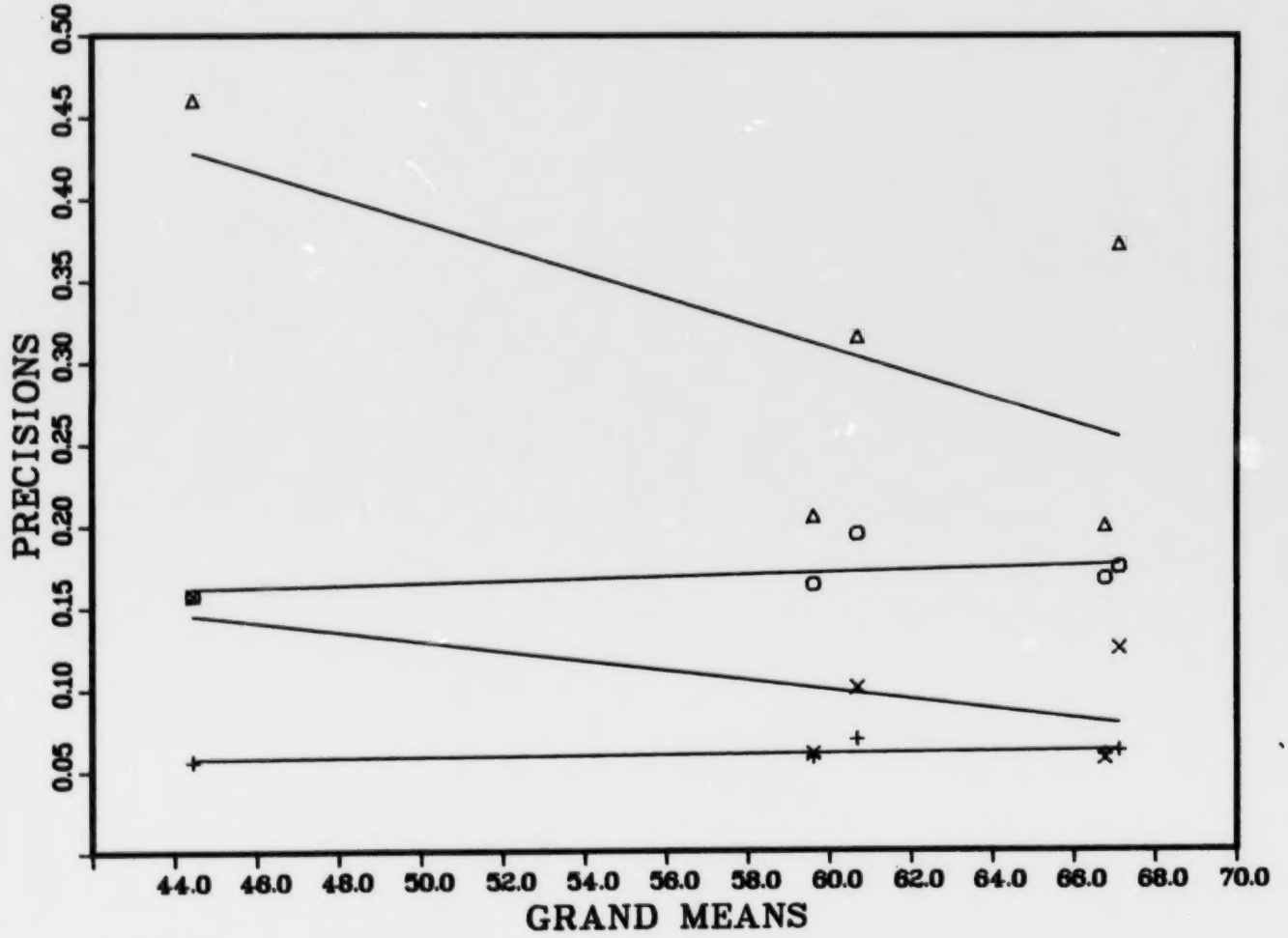

Figure .3.b

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E

APPENDIX 1a

Detailed computation of analytical data of Fe-total by Ag-reduction method

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 1. BACKGRUVAN 79-3

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	44.9000	44.9400	44.91500	.9000E-03
		2	44.9400	44.8800		
2	AU 2	1	44.7731	44.7969	44.74908	.1910E-02
		2	44.7251	44.7012		
3	AU 3	1	45.3300	45.3300	45.27000	.1440E-01
		2	45.3300	45.0900		
4	AU 4	1	44.9923	44.9770	44.96253	.7738E-03
		2	44.9293	44.9515		
5	AU 5	1	45.3902	45.1944	45.29788	.1145E-01
		2	45.3902	45.2167		
6	AU 6	1	45.0230	44.9762	44.97265	.1431E-02
		2	44.9336	44.9578		
7	AU 7	1	44.5900	44.6200	45.09500	.3204E+00
		2	45.6000	45.5700		
8	CND 1	1	44.5300	44.3700	44.43250	.6025E-02
		2	44.4600	44.3700		
9	CND 2	1	44.7200	44.8600	44.70250	.3629E-01
		2	44.4300	44.8000		
10	CND 3	1	44.9400	45.4000	45.23250	.8049E-01
		2	45.0500	45.5400		
11	CND 4	1	44.5300	44.4100	44.51500	.1177E-01
		2	44.6600	44.4600		
12	FR 1	1	45.0523	45.0058	45.01740	.1986E-02
		2	45.0523	44.9592		
13	FR 2	1	44.9033	44.9964	44.92660	.7938E-02
		2	44.9964	44.8103		
14	FR 3	1	44.9767	45.0900	45.05920	.3312E-01
		2	45.2975	44.8726		
15	FR 4	1	45.2480	45.2242	45.15888	.9186E-02
		2	45.1230	45.0403		
16	FR 5	1	44.8113	44.8471	44.84630	.1244E-02
		2	44.8324	44.8944		
17	GER 1	1	44.7000	45.0200	44.89250	.1929E-01
		2	44.8900	44.9600		
18	IND 1	1	45.1700	44.4900	44.77750	.1512E+00
		2	45.0500	44.4000		
19	IND 2	1	44.8600	44.8300	44.85000	.2000E-03
		2	44.8600	44.8500		
20	IT 1	1	44.5700	44.4500	44.45500	.6433E-02
		2	44.4000	44.4000		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 1. BACKGRUVAN 79-3

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
21	IT 2	1	44.5500	44.5000	44.52500	.4167E-02
		2	44.6000	44.4500		
22	JP 1	1	44.8730	44.6910	44.79375	.6798E-02
		2	44.7650	44.8460		
23	JP 2	1	45.1820	45.1450	45.14500	.9127E-03
		2	45.1080	45.1450		
24	JP 3	1	45.0920	44.9520	45.02825	.3379E-02
		2	45.0440	45.0250		
25	JP 4	1	45.0070	45.0580	45.01950	.8870E-03
		2	44.9880	45.0250		
26	JP 5	1	44.9600	44.9400	44.94250	.9583E-03
		2	44.9000	44.9700		
27	JP 6	1	44.8210	44.7730	44.84275	.3512E-02
		2	44.9110	44.8660		
28	JP 7	1	45.1500	44.9470	45.07325	.8069E-02
		2	45.1220	45.0740		
29	SW 1	1	45.0200	45.0800	45.01500	.2833E-02
		2	44.9500	45.0100		
30	SW 2	1	44.7700	44.7900	44.80750	.1092E-02
		2	44.8300	44.8400		
31	SW 3	1	44.9500	44.8600	44.85000	.1420E-01
		2	44.9100	44.6800		
32	USA 1	1	44.8700	45.0100	45.00000	.8667E-02
		2	45.0300	45.0900		
33	USA 2	1	45.1600	45.1900	45.14250	.2158E-02
		2	45.1400	45.0800		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 1. BACKGRUVAN 79-3

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	90.440
CHI-SQUARE(.95,61)	80.229

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR BOTTLE VARIANCE

LAB-18	BOTTLE- 1	C-CALC =	.204	C-CRIT(2,62) =	.167
LAB-18	BOTTLE- 2	C-CALC =	.234	C-CRIT(2,61) =	.170
LAB-10	BOTTLE- 2	C-CALC =	.173	C-CRIT(2,60) =	.172
LAB-10	BOTTLE- 1	C-CALC =	.185	C-CRIT(2,59) =	.174
LAB-14	BOTTLE- 2	C-CALC =	.193	C-CRIT(2,58) =	.177
LAB- 9	BOTTLE- 2	C-CALC =	.182	C-CRIT(2,57) =	.179

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 3	ZERO WITHIN-BOTTLE VARIANCE
LAB- 9	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB-10	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB-14	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB-18	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB-20	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 1. BACKGRUVAN 79-3

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	26	.41729180E+01	.16049685E+00
BETWEEN BOTTLES	27	.10644806E+01	.39425207E-01
WITHIN BOTTLES	54	.29201921E+00	.54077631E-02
TOTAL	107	.55294178E+01	

F-CALC = 7.290 F-CRIT(.95;27,54) = 1.693

BETWEEN-BOTTLE VARIANCE IS SIGNIFICANT
TEST SAMPLE IS PRESUMED TO BE INHOMOGENEOUS

ONE-WAY ANOVA CAN NOT BE PERFORMED
PRECISION ESTIMATES FROM TWO-WAY ANOVA

REPEATABILITY VARIANCE	.54077631E-02
REPEATABILITY STD. DEV.	.07353749
BETWEEN-LABORATORIES VARIANCE	.30267910E-01
BETWEEN-LABORATORIES STD. DEV.	.17397675
REPEATABILITY	.20811111
PERMISSIBLE TOLERANCE BETWEEN LABS	.51387526
GRAND MEAN	44.91912222
VARIANCE OF THE GRAND MEAN	.14860819E-02

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	66.8900	67.0000	67.04750	.1663E-01
		2	67.1200	67.1800		
2	AU 2	1	66.8101	66.8807	66.82553	.2139E-02
		2	66.7714	66.8399		
3	AU 3	1	67.0200	67.2000	67.08500	.7300E-02
		2	67.0200	67.1000		
4	AU 4	1	67.0386	67.1130	67.11582	.3079E-02
		2	67.1503	67.1614		
5	AU 5	1	67.2089	67.1442	67.17845	.3593E-02
		2	67.2462	67.1145		
6	AU 6	1	66.9018	66.8963	66.99450	.1224E-01
		2	67.0783	67.1016		
7	AU 7	1	67.1400	67.3300	67.11750	.2982E-01
		2	66.9100	67.0900		
8	CND 1	1	67.2200	67.2000	67.22250	.1625E-02
		2	67.2800	67.1900		
9	CND 2	1	67.1100	67.4000	66.98750	.1100E+00
		2	66.7500	66.6900		
10	CND 3	1	67.1800	67.2200	67.15000	.4667E-02
		2	67.0600	67.1400		
11	CND 4	1	66.8600	66.8300	66.87750	.3225E-02
		2	66.8600	66.9600		
12	FR 1	1	67.1131	67.0199	67.06470	.2894E-02
		2	67.1094	67.0164		
13	FR 2	1	67.0572	66.9641	66.94083	.7945E-02
		2	66.8710	66.8710		
14	FR 3	1	67.0070	67.0491	67.04395	.1789E-02
		2	67.0182	67.1015		
15	FR 4	1	67.3145	67.2002	67.26785	.3870E-02
		2	67.2302	67.3265		
16	FR 5	1	67.1087	67.3742	67.25015	.1204E-01
		2	67.2419	67.2758		
17	GER 1	1	67.3300	67.1800	67.13750	.3522E-01
		2	66.8800	67.1600		
18	IND 1	1	67.0300	67.2000	67.14000	.1607E-01
		2	67.0400	67.2900		
19	IND 2	1	66.9600	66.9900	66.94250	.2625E-02
		2	66.8700	66.9500		
20	IT 1	1	67.1000	67.0800	67.16250	.7892E-02
		2	67.2000	67.2700		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
21	IT 2	1	67.2000	67.3000	67.20750	.4225E-02
		2	67.1800	67.1500		
22	JP 1	1	67.2060	67.1800	67.20325	.7209E-03
		2	67.2400	67.1870		
23	JP 2	1	67.2060	67.2060	67.21525	.3423E-03
		2	67.2060	67.2430		
24	JP 3	1	67.1090	67.0940	67.07725	.3814E-02
		2	67.1200	66.9860		
25	JP 4	1	67.0340	67.0420	67.00750	.1364E-02
		2	66.9640	66.9900		
26	JP 5	1	66.9200	66.9600	66.94250	.4250E-03
		2	66.9600	66.9300		
27	JP 6	1	67.1170	67.1130	67.11225	.2092E-04
		2	67.1060	67.1130		
28	JP 7	1	67.0760	67.1710	67.15875	.4074E-02
		2	67.2310	67.1570		
29	SW 1	1	66.7300	66.7600	66.71250	.1558E-02
		2	66.6800	66.6800		
30	SW 2	1	67.0200	66.8000	66.93500	.8967E-02
		2	66.9700	66.9500		
31	SW 3	1	67.4600	67.2100	67.26750	.3149E-01
		2	67.0500	67.3500		
32	USA 1	1	67.0200	67.0600	67.07000	.1533E-02
		2	67.0900	67.1100		
33	USA 2	1	66.8100	66.8200	66.91500	.2550E-01
		2	67.1500	66.8800		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	66.450
CHI-SQUARE(.95,59)	77.927

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB-13	ZERO WITHIN-BOTTLE VARIANCE
LAB-23	ZERO WITHIN-BOTTLE VARIANCE
LAB-29	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG178 FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	29	.16186834E+01	.55816670E-01
BETWEEN BOTTLES	30	.61308226E+00	.20436075E-01
WITHIN BOTTLES	60	.46356776E+00	.77261293E-02
TOTAL	119	.26953335E+01	

F-CALC = 2.645 F-CRIT(.95;30,60) = 1.649

BETWEEN-BOTTLE VARIANCE IS SIGNIFICANT
TEST SAMPLE IS PRESUMED TO BE INHOMOGENEOUS

ONE-WAY ANOVA CAN NOT BE PERFORMED
PRECISION ESTIMATES FROM TWO-WAY ANOVA

REPEATABILITY VARIANCE	.77261293E-02
REPEATABILITY STD. DEV.	.08789840
BETWEEN-LABORATORIES VARIANCE	.88451487E-02
BETWEEN-LABORATORIES STD. DEV.	.09404865
REPEATABILITY	.24875248
PERMISSIBLE TOLERANCE BETWEEN LABS	.31902791
GRAND MEAN	67.08358167
VARIANCE OF THE GRAND MEAN	.46513892E-03

ISO/TC102/SC2/WG17? FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. MARCONA JSS 850-1

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	66.5400	66.7300	66.67000	.7667E-02
		2	66.7100	66.7000		
2	AU 2	1	66.8479	66.8357	66.80043	.2376E-02
		2	66.7692	66.7489		
3	AU 3	1	66.7400	66.8300	66.80750	.2025E-02
		2	66.8300	66.8300		
4	AU 4	1	66.7680	66.6000	66.69845	.1473E-01
		2	66.8338	66.5920		
5	AU 5	1	66.9483	66.8243	66.86075	.7274E-02
		2	66.9111	66.7593		
6	AU 6	1	66.6866	66.7453	66.75208	.2395E-02
		2	66.7992	66.7772		
7	AU 7	1	66.7400	66.9300	66.74250	.1909E-01
		2	66.6000	66.7000		
8	CND 1	1	66.7600	66.8300	66.77000	.3000E-02
		2	66.7000	66.7900		
9	CND 2	1	66.9800	66.9500	66.93750	.1225E-02
		2	66.9000	66.9200		
10	CND 3	1	66.9900	66.8900	66.94750	.3758E-02
		2	67.0100	66.9000		
11	CND 4	1	66.5600	66.7700	66.68500	.1070E-01
		2	66.6400	66.7700		
12	FR 1	1	66.6476	66.7407	66.62258	.8275E-02
		2	66.5510	66.5510		
13	FR 2	1	66.8710	66.8710	66.82445	.2889E-02
		2	66.7779	66.7779		
14	FR 3	1	66.4975	66.8372	66.65638	.2669E-01
		2	66.5408	66.7500		
15	FR 4	1	66.8980	66.9247	66.92888	.3459E-02
		2	66.8801	67.0127		
16	FR 5	1	66.7983	66.9539	66.92658	.1674E-01
		2	66.8589	67.0952		
17	GER 1	1	66.6600	66.8500	66.57250	.1321E+00
		2	66.0400	66.7400		
18	IND 1	1	67.0100	66.9200	67.00250	.3492E-02
		2	67.0600	67.0200		
19	IND 2	1	66.5100	66.5300	66.54000	.1200E-02
		2	66.5900	66.5300		
20	IT 1	1	66.8500	66.8500	66.83000	.6000E-03
		2	66.8000	66.8200		

ISO/TC102/SC2/WG178 FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. MARCONA JSS 850-1

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
21	IT 2	1	66.8500	66.8500	66.85500	.2433E-02
		2	66.8000	66.9200		
22	JP 1	1	66.8270	66.8820	66.84175	.9136E-03
		2	66.8120	66.8460		
23	JP 2	1	66.8330	66.8710	66.83325	.9376E-03
		2	66.8330	66.7960		
24	JP 3	1	66.7860	66.7000	66.73325	.1500E-02
		2	66.7380	66.7090		
25	JP 4	1	66.6440	66.7330	66.66150	.2692E-02
		2	66.6100	66.6590		
26	JP 5	1	66.6200	66.7900	66.71750	.5292E-02
		2	66.7100	66.7500		
27	JP 6	1	66.7200	66.7770	66.76375	.8543E-03
		2	66.7810	66.7770		
28	JP 7	1	66.8130	66.7420	66.76825	.3045E-02
		2	66.7030	66.8150		
29	SW 1	1	67.0100	66.8600	66.94000	.9800E-02
		2	67.0400	66.8500		
30	SW 2	1	66.5700	66.6400	66.66000	.9667E-02
		2	66.6300	66.8000		
31	SW 3	1	66.9200	66.8700	66.81000	.1047E-01
		2	66.7500	66.7000		
32	USA 1	1	66.6800	66.6300	66.67500	.2300E-02
		2	66.6500	66.7400		
33	USA 2	1	66.7300	66.8100	66.71500	.8367E-02
		2	66.5900	66.7300		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. MARCONA JSS 850-1

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	60.172
CHI-SQUARE(.95,55)	73.308

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 3	ZERO WITHIN-BOTTLE VARIANCE
LAB-12	ZERO WITHIN-BOTTLE VARIANCE
LAB-13	ZERO WITHIN-BOTTLE VARIANCE
LAB-20	ZERO WITHIN-BOTTLE VARIANCE
LAB-21	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. MARCONA JSS 850-1

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	27	.15216216E+01	.56356355E-01
BETWEEN BOTTLES	28	.27639049E+00	.98710890E-02
WITHIN BOTTLES	56	.65875478E+00	.11763478E-01
TOTAL	111	.24567668E+01	

F-CALC = .839 F-CRIT(.95;28,56) = 1.678

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. MARCONA JSS 850-1

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	74.691
CHI-SQUARE(.95,32)	46.201

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-17 C-CALC = .403 C-CRIT(4,33) = .147

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB-17 COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. MARCONA JSS 850-1

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	31	.14994569E+01	.48369579E-01
WITHIN LABS	96	.58753861E+00	.61201938E-02
TOTAL	127	.20869956E+01	

REPEATABILITY VARIANCE	.61201938E-02
REPEATABILITY STD. DEV.	.07823167
BETWEEN-LABORATORIES VARIANCE	.10562346E-01
BETWEEN-LABORATORIES STD. DEV.	.10277328
REPEATABILITY	.22139562
PERMISSIBLE TOLERANCE BETWEEN LABS	.33030408
GRAND MEAN	66.78054062
VARIANCE OF THE GRAND MEAN	.37788734E-03

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	59.4800	59.4400	59.52750	.6492E-02
		2	59.5800	59.6100		
2	AU 2	1	59.5793	59.4189	59.50668	.4947E-02
		2	59.5438	59.4847		
3	AU 3	1	59.6100	59.7600	59.66250	.5025E-02
		2	59.6700	59.6100		
4	AU 4	1	59.5336	59.4580	59.51073	.2621E-02
		2	59.5710	59.4803		
5	AU 5	1	59.7995	59.6108	59.70200	.1862E-01
		2	59.8367	59.5610		
6	AU 6	1	59.8131	59.6306	59.69538	.8870E-02
		2	59.7287	59.6091		
7	AU 7	1	59.4800	59.6100	59.49750	.1123E-01
		2	59.3600	59.5400		
8	CND 1	1	59.8900	59.7200	59.82250	.1062E-01
		2	59.9300	59.7500		
9	CND 2	1	59.7100	59.7400	59.61750	.3963E-01
		2	59.7000	59.3200		
10	CND 3	1	59.6100	59.6900	59.67750	.2492E-02
		2	59.6800	59.7300		
11	CND 4	1	59.5900	59.6200	59.58250	.8250E-03
		2	59.5600	59.5600		
12	FR 1	1	59.7595	59.7595	59.66480	.1196E-01
		2	59.5701	59.5701		
13	FR 2	1	59.5174	59.5174	59.49413	.2167E-02
		2	59.4243	59.5174		
14	FR 3	1	59.6439	59.6259	59.68938	.4185E-02
		2	59.7600	59.7277		
15	FR 4	1	59.7341	59.7024	59.64660	.8513E-02
		2	59.6224	59.5275		
16	FR 5	1	59.8875	59.8194	59.82525	.3322E-02
		2	59.8443	59.7498		
17	GFR 1	1	59.4600	59.6500	59.57250	.7492E-02
		2	59.5500	59.6300		
18	IND 1	1	59.6900	59.4800	59.57250	.7625E-02
		2	59.5500	59.5700		
19	IND 2	1	59.4400	59.4700	59.45000	.3333E-03
		2	59.4300	59.4600		
20	IT 1	1	59.5300	59.6800	59.69000	.1380E-01
		2	59.8000	59.7500		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
21	IT 2	1	59.6500	59.8000	59.74250	.6558E-02
		2	59.8200	59.7000		
22	JP 1	1	59.6460	59.7200	59.67750	.9537E-03
		2	59.6730	59.6710		
23	JP 2	1	59.5730	59.6660	59.59625	.2162E-02
		2	59.5730	59.5730		
24	JP 3	1	59.5110	59.5530	59.52075	.2096E-02
		2	59.4600	59.5590		
25	JP 4	1	59.5070	59.5740	59.54800	.1027E-02
		2	59.5730	59.5380		
26	JP 5	1	59.6100	59.7100	59.66750	.5492E-02
		2	59.6000	59.7500		
27	JP 6	1	59.5060	59.5540	59.52700	.4920E-03
		2	59.5120	59.5360		
28	JP 7	1	59.5450	59.4890	59.56850	.5532E-02
		2	59.5730	59.6670		
29	SW 1	1	60.1700	60.2500	60.20000	.1467E-02
		2	60.1700	60.2100		
30	SW 2	1	59.3900	59.5000	59.45250	.1169E-01
		2	59.3400	59.5800		
31	SW 3	1	59.6500	59.6400	59.88250	.2419E+00
		2	59.6200	60.6200		
32	USA 1	1	59.6700	59.5400	59.58500	.4700E-02
		2	59.5200	59.6100		
33	USA 2	1	59.5800	59.7000	59.61250	.3425E-02
		2	59.5800	59.5900		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	76.072
CHI-SQUARE(.95,57)	75.620

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR BOTTLE VARIANCE

LAB-31 BOTTLE- 2 C-CALC = .538 C-CRIT(2,58) = .177

DOORNBOS' TEST FOR BOTTLE MEANS

LAB-29	BOTTLE- 1	T-CALC = 4.130	T-CRIT(.00076,64) = 3.538
LAB-29	BOTTLE- 2	T-CALC = 4.660	T-CRIT(.00077,63) = 3.536
LAB-31	BOTTLE- 2	T-CALC = 4.836	T-CRIT(.00078,62) = 3.533

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB-11	ZERO WITHIN-BOTTLE VARIANCE
LAB-12	ZERO WITHIN-BOTTLE VARIANCE
LAB-13	ZERO WITHIN-BOTTLE VARIANCE
LAB-23	ZERO WITHIN-BOTTLE VARIANCE
LAB-29	DOORNBOS' TEST FOR BOTTLE MEANS
LAB-31	DOORNBOS' TEST FOR BOTTLE MEANS

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	26	.10719207E+01	.41227718E-01
BETWEEN BOTTLES	27	.16794712E+00	.62202639E-02
WITHIN BOTTLES	54	.42537425E+00	.78773009E-02
TOTAL	107	.16652420E+01	

F-CALC = .790 F-CRIT(.95;27,54) = 1.693

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC 96.098
CHI-SQUARE(.95,32) 46.201

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-31 C-CALC = .528 C-CRIT(4,33) = .147

BOTTLE- 2 REPLICATE- 2 DIXON'S TEST WITHIN LAB-31
R10 = .970 D-CRIT(4) = .829

LAB- 9 C-CALC = .183 C-CRIT(4,33) = .147

BOTTLE- 2 REPLICATE- 2 DIXON'S TEST WITHIN LAB- 9
R10 = .905 D-CRIT(4) = .829

NONE REJECTED

DIXON'S TEST FOR LABORATORY MEANS

LAB-29 R = .535 D-CRIT(33) = .397

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB-29 DIXON'S TEST FOR LABORATORY MEANS

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

LAB- 9	BOTTLE- 2	REPLICATE- 2	59.3200
LAB-31	BOTTLE- 2	REPLICATE- 2	60.6200

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 4. LABRADOR NBS 692

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	31	.11778977E+01	.37996699E-01
WITHIN LABS	94	.52711452E+00	.56076013E-02
TOTAL	125	.17050122E+01	

REPEATABILITY VARIANCE	.56076013E-02
REPEATABILITY STD. DEV.	.07488392
BETWEEN-LABORATORIES VARIANCE	.82268055E-02
BETWEEN-LABORATORIES STD. DEV.	.09070174
REPEATABILITY	.21192149
PERMISSIBLE TOLERANCE BETWEEN LABS	.29722554
GRAND MEAN	59.61279127
VARIANCE OF THE GRAND MEAN	.30256405E-03

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 3. CANADA SCH-1

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	60.2800	60.3700	60.43750	.1989E-01
		2	60.6000	60.5000		
2	AU 2	1	60.7501	60.6443	60.67683	.5266E-02
		2	60.5911	60.7218		
3	AU 3	1	60.7000	60.5900	60.63750	.5358E-02
		2	60.7000	60.5600		
4	AU 4	1	60.5042	60.4483	60.50073	.1494E-02
		2	60.5414	60.5090		
5	AU 5	1	60.6559	60.7354	60.66570	.4219E-02
		2	60.5814	60.6901		
6	AU 6	1	60.7454	60.6284	60.65000	.4098E-02
		2	60.6117	60.6145		
7	AU 7	1	60.7700	60.9200	60.85250	.7092E-02
		2	60.7900	60.9300		
8	CND 1	1	60.8000	60.8400	60.80250	.1492E-02
		2	60.7500	60.8200		
9	CND 2	1	60.7000	60.5800	60.72000	.1333E-01
		2	60.7400	60.8600		
10	CND 3	1	60.6800	60.6700	60.71750	.2425E-02
		2	60.7600	60.7600		
11	CND 4	1	60.5900	60.6800	60.60500	.2967E-02
		2	60.5500	60.6000		
12	FR 1	1	60.8299	60.7369	60.77713	.2939E-02
		2	60.8174	60.7243		
13	FR 2	1	60.6344	60.7275	60.68095	.2889E-02
		2	60.6344	60.7275		
14	FR 3	1	60.6591	60.8801	60.76528	.8402E-02
		2	60.7798	60.7421		
15	FR 4	1	60.8702	60.6784	60.82215	.1124E-01
		2	60.9256	60.8144		
16	FR 5	1	60.8336	60.7336	60.78650	.3565E-02
		2	60.7362	60.8426		
17	GER 1	1	60.1100	60.5000	60.35750	.2903E-01
		2	60.4100	60.4100		
18	IND 1	1	60.5000	60.5000	60.41250	.1036E-01
		2	60.3400	60.3100		
19	IND 2	1	60.3700	60.3200	60.37000	.1400E-02
		2	60.3800	60.4100		
20	IT 1	1	60.6800	60.7100	60.72750	.2625E-02
		2	60.7200	60.8000		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 5. CANADA SC4-1

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
21	IT 2	1	60.7500	60.6200	60.73500	.8967E-02
		2	60.7200	60.8500		
22	JP 1	1	60.7540	60.8160	60.75050	.2358E-02
		2	60.7020	60.7300		
23	JP 2	1	60.6900	60.5970	60.67600	.3078E-02
		2	60.7270	60.6900		
24	JP 3	1	60.5810	60.5750	60.59350	.8870E-03
		2	60.6380	60.5800		
25	JP 4	1	60.5240	60.5490	60.53525	.2349E-02
		2	60.5920	60.4760		
26	JP 5	1	60.6400	60.7100	60.69750	.1558E-02
		2	60.7100	60.7300		
27	JP 6	1	60.5960	60.6530	60.63575	.7343E-03
		2	60.6410	60.6530		
28	JP 7	1	60.7590	60.7030	60.72000	.8147E-03
		2	60.7230	60.6950		
29	SW 1	1	60.7400	60.7700	60.72500	.1367E-02
		2	60.6900	60.7000		
30	SW 2	1	60.5300	60.6900	60.58000	.6067E-02
		2	60.5200	60.5800		
31	SW 3	1	60.7400	60.6100	60.66250	.1109E-01
		2	60.5400	60.7600		
32	USA 1	1	60.4000	60.4600	60.50500	.8367E-02
		2	60.5600	60.6000		
33	USA 2	1	60.5600	60.6000	60.59250	.2492E-02
		2	60.6600	60.5500		

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 5. CANADA SCH-1

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	36.875
CHI-SQUARE(.95,59)	77.927

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

COORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB-10	ZERO WITHIN-BOTTLE VARIANCE
LAB-17	ZERO WITHIN-BOTTLE VARIANCE
LAB-18	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 5. CANADA SCH-1

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	29	.14983605E+01	.51667605E-01
BETWEEN BOTTLES	30	.16465905E+00	.54886352E-02
WITHIN BOTTLES	60	.28055564E+00	.46759273E-02
TOTAL	119	.19435752E+01	

F-CALC = 1.174 F-CRIT(.95;30,60) = 1.649

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG17B FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 5. CANADA SCH-1

PARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	37.512
CHI-SQUARE(.95,32)	46.201

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG17 FE-TOTAL SILVER REDUCTION METHOD DOC. ISO/TC102/SC2 N571E
SAMPLE: 5. CANADA SCH-1

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	32	.21036226E+01	.65738207E-01
WITHIN LABS	99	.57063969E+00	.57640373E-02
TOTAL	131	.26742623E+01	

REPEATABILITY VARIANCE	.57640373E-02
REPEATABILITY STD. DEV.	.07592126
BETWEEN-LABORATORIES VARIANCE	.14993542E-01
BETWEEN-LABORATORIES STD. DEV.	.12244812
REPEATABILITY	.21485716
PERMISSIBLE TOLERANCE BETWEEN LABS	.37836964
GRAND MEAN	60.64768939
VARIANCE OF THE GRAND MEAN	.49801672E-03

APPENDIX 1b

Detailed computation of analytical data of

Fe-total by $TiCl_3$-reduction method

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 1. BACKKGRUVAN 79-3

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	44.8000	43.7700	44.23750	.2615E+00
		2	44.5400	43.8400		
2	AU 2	1	44.3565	44.2867	44.33013	.9762E-03
		2	44.3487	44.3286		
3	AU 3	1	44.6213	44.6800	44.63435	.1170E-02
		2	44.5990	44.6371		
4	FR 1	1	44.2238	44.3984	44.34603	.6910E-02
		2	44.3635	44.3984		
5	FR 2	1	44.2611	44.2611	44.20875	.4466E-02
		2	44.1913	44.1215		
6	FR 3	1	44.7429	44.3306	44.51865	.3024E-01
		2	44.5476	44.4535		
7	FR 4	1	44.5069	44.4153	44.47180	.2217E-02
		2	44.5141	44.4509		
8	FR 5	1	44.5383	44.5350	44.51533	.6508E-03
		2	44.5020	44.4860		
9	GER 1	1	44.3600	44.3600	44.43750	.1283E-01
		2	44.6000	44.4300		
10	IT 1	1	44.6100	44.5300	44.55500	.3767E-02
		2	44.4800	44.6000		
11	IT 2	1	44.5800	44.6200	44.61250	.6492E-02
		2	44.5300	44.7200		
12	JP 1	1	44.5680	44.5400	44.54375	.5789E-03
		2	44.5550	44.5120		
13	JP 2	1	44.5660	44.4960	44.51350	.1225E-02
		2	44.4960	44.4960		
14	JP 2	1	44.3980	44.3690	44.39050	.1299E-02
		2	44.3570	44.4380		
15	JP 4	1	44.6080	44.5660	44.56250	.1094E-02
		2	44.5380	44.5380		
16	JP 5	1	44.3300	44.3700	44.38000	.1467E-02
		2	44.4100	44.4100		
17	JP 6	1	44.4760	44.5340	44.52500	.1127E-02
		2	44.5380	44.5520		
18	JP 7	1	44.6330	44.6610	44.65925	.3509E-03
		2	44.6660	44.6770		
19	US 1	1	44.0600	44.1300	44.04000	.6200E-02
		2	44.0300	43.9400		

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 1. BACKKGRUVAN 79-3

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	61.007
CHI-SQUARE(.95,27)	40.119

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR BOTTLE VARIANCE

LAB- 1	BOTTLE- 1	C-CALC =	.567	C-CRIT(2,28) =	.308
LAB- 1	BOTTLE- 2	C-CALC =	.605	C-CRIT(2,27) =	.316
LAB- 6	BOTTLE- 1	C-CALC =	.532	C-CRIT(2,26) =	.325

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 1	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB- 5	ZERO WITHIN-BOTTLE VARIANCE
LAB- 6	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB- 9	ZERO WITHIN-BOTTLE VARIANCE
LAB-13	ZERO WITHIN-BOTTLE VARIANCE
LAB-15	ZERO WITHIN-BOTTLE VARIANCE
LAB-16	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 1. BACKKGRUVAN 79-3

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	11	.13080485E+01	.11891350E+00
BETWEEN BOTTLES	12	.24950965E-01	.20792471E-02
WITHIN BOTTLES	24	.70264107E-01	.29276711E-02
TOTAL	47	.14032635E+01	

F-CALC = .710 F-CRIT(.95;12,24) = 2.183

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 1. BACKKGRUVAN 79-3

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	94.321
CHI-SQUARE(.95,18)	28.877

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 1	C-CALC =	.759	C-CRIT(4,19) =	.229
LAB- 6	C-CALC =	.364	C-CRIT(4,18) =	.239

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 1 COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB- 6 COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 1. BACKKGRUVAN 79-3

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	16	.16431485E+01	.10269678E+00
WITHIN LABS	51	.15844618E+00	.31067879E-02
TOTAL	67	.18015947E+01	

REPEATABILITY VARIANCE	.31067879E-02
REPEATABILITY STD. DEV.	.05573857
BETWEEN-LABORATORIES VARIANCE	.24897498E-01
BETWEEN-LABORATORIES STD. DEV.	.15778941
REPEATABILITY	.15774015
PERMISSIBLE TOLERANCE BETWEEN LABS	.46026357
GRAND MEAN	44.45446324
VARIANCE OF THE GRAND MEAN	.15102468E-02

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	66.9900	66.9900	67.01500	.5100E-02
		2	66.9600	67.1200		
2	AU 2	1	67.0164	67.0108	67.02198	.2285E-03
		2	67.0443	67.0164		
3	AU 3	1	67.0618	67.1596	67.13430	.2350E-02
		2	67.1623	67.1535		
4	FR 1	1	67.0164	67.0164	67.01640	0.
		2	67.0164	67.0164		
5	FR 2	1	67.0898	67.0898	67.08980	.3248E-02
		2	67.0200	67.1596		
6	FR 3	1	67.5364	67.3176	67.44213	.2082E-01
		2	67.5944	67.3201		
7	FR 4	1	67.2126	67.1985	67.17250	.4460E-02
		2	67.2062	67.0727		
8	FR 5	1	67.0673	67.1640	67.15113	.4250E-02
		2	67.2256	67.1476		
9	GER 1	1	67.4200	67.6800	67.55500	.2090E-01
		2	67.4400	67.6800		
10	IT 1	1	67.2000	67.2800	67.23250	.4892E-02
		2	67.3000	67.1500		
11	IT 2	1	67.0500	67.1600	67.16000	.8067E-02
		2	67.2700	67.1600		
12	JP 1	1	67.1790	67.1120	67.14750	.1847E-02
		2	67.1900	67.1090		
13	JP 2	1	67.0860	67.1140	67.07350	.1067E-02
		2	67.0470	67.0470		
14	JP 2	1	67.2400	67.2930	67.26350	.5403E-03
		2	67.2510	67.2700		
15	JP 4	1	67.0160	67.0860	67.05800	.1176E-02
		2	67.0440	67.0860		
16	JP 5	1	66.8200	66.8800	66.84000	.2400E-02
		2	66.7800	66.8800		
17	JP 6	1	67.0820	66.9490	67.02450	.3310E-02
		2	67.0140	67.0530		
18	JP 7	1	67.1290	67.0400	67.06900	.1894E-02
		2	67.0730	67.0340		
19	US 1	1	67.0300	67.0000	67.02500	.2300E-02
		2	67.0900	66.9800		

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	25.485
CHI-SQUARE(.95,29)	42.565

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 1	ZERO WITHIN-BOTTLE VARIANCE
LAB- 4	ZERO WITHIN-BOTTLE VARIANCE
LAB- 5	ZERO WITHIN-BOTTLE VARIANCE
LAB-13	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG168 FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.17146160E+01	.12247257E+00
BETWEEN BOTTLES	15	.27584573E-01	.18389716E-02
WITHIN BOTTLES	30	.21072897E+00	.70242990E-02
TOTAL	59	.19529296E+01	

F-CALC = .262 F-CRIT(.95;15,30) = 2.015

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	27.097
CHI-SQUARE(.95,17)	27.593

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

LAB- 9 R = .541 D-CRIT(19) = .501

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 9 DIXON'S TEST FOR LABORATORY MEANS

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 2. SAVAGE JSS 852-1 76-17

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	17	.11118425E+01	.65402502E-01
WITHIN LABS	54	.20385862E+00	.37751597E-02
TOTAL	71	.13157012E+01	

REPEATABILITY VARIANCE	.37751597E-02
REPEATABILITY STD. DEV.	.06144233
BETWEEN-LABORATORIES VARIANCE	.15406836E-01
BETWEEN-LABORATORIES STD. DEV.	.12412427
REPEATABILITY	.17388179
PERMISSIBLE TOLERANCE BETWEEN LABS	.37216830
GRAND MEAN	67.10759583
VARIANCE OF THE GRAND MEAN	.90836808E-03

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 3. MARCONA JSS 850-1

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	66.7400	66.7400	66.74750	.2250E-03
		2	66.7400	66.7700		
2	AU 2	1	66.7372	66.6953	66.72400	.1114E-02
		2	66.7651	66.6984		
3	AU 3	1	66.8059	66.8029	66.78098	.2702E-02
		2	66.8119	66.7032		
4	FR 1	1	66.7372	66.7372	66.71973	.1222E-02
		2	66.6673	66.7372		
5	FR 2	1	66.8105	66.8803	66.86285	.1218E-02
		2	66.8803	66.8803		
6	FR 3	1	66.8515	66.7767	66.72488	.1270E-01
		2	66.5941	66.6772		
7	FR 4	1	66.7830	66.6814	66.76070	.3677E-02
		2	66.8254	66.7530		
8	FR 5	1	66.9108	66.7862	66.83283	.2940E-02
		2	66.8232	66.8111		
9	GER 1	1	66.6600	66.8200	66.74000	.8533E-02
		2	66.6600	66.8200		
10	IT 1	1	66.7500	66.7000	66.71500	.9667E-03
		2	66.6800	66.7300		
11	IT 2	1	66.8200	66.9400	66.88000	.4800E-02
		2	66.8200	66.9400		
12	JP 1	1	66.7810	66.7680	66.75200	.8847E-03
		2	66.7130	66.7460		
13	JP 2	1	66.6950	66.7650	66.78200	.4422E-02
		2	66.8340	66.8340		
14	JP 2	1	66.7910	66.8360	66.83300	.8647E-03
		2	66.8580	66.8470		
15	JP 4	1	66.6670	66.7510	66.72650	.2401E-02
		2	66.7090	66.7790		
16	JP 5	1	66.7200	66.7500	66.71500	.4433E-02
		2	66.6200	66.7700		
17	JP 6	1	66.7200	66.7740	66.73325	.1172E-02
		2	66.6940	66.7450		
18	JP 7	1	66.7000	66.7100	66.75275	.3217E-02
		2	66.8160	66.7850		
19	US 1	1	66.6100	66.6900	66.59750	.8492E-02
		2	66.4700	66.6200		

ISO/TC102/SC2/WG168 FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 3. MARCONA JSS 850-1

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	22.218
CHI-SQUARE(.95,29)	42.565

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 1	ZERO WITHIN-BOTTLE VARIANCE
LAB- 4	ZERO WITHIN-BOTTLE VARIANCE
LAB- 5	ZERO WITHIN-BOTTLE VARIANCE
LAB-13	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 3. MARCONA JSS 850-1

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.23863322E+00	.17045230E-01
BETWEEN BOTTLES	15	.66183895E-01	.44122597E-02
WITHIN BOTTLES	30	.11050187E+00	.36833956E-02
TOTAL	59	.41531899E+00	

F-CALC = 1.198 F-CRIT(.95;15,30) = 2.015

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG169 FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 3. MARCONA JSS 850-1

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	21.476
CHI-SQUARE(.95,18)	28.877

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 3. MARCONA JSS 850-1

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	18	.29386848E+00	.16326027E-01
WITHIN LABS	57	.19794530E+00	.34727246E-02
TOTAL	75	.49181378E+00	

REPEATABILITY VARIANCE	.34727246E-02
REPEATABILITY STD. DEV.	.05892983
BETWEEN-LABORATORIES VARIANCE	.32133255E-02
BETWEEN-LABORATORIES STD. DEV.	.05668620
REPEATABILITY	.16677141
PERMISSIBLE TOLERANCE BETWEEN LABS	.19910187
GRAND MEAN	66.75686579
VARIANCE OF THE GRAND MEAN	.21481614E-03

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 4. LABRADOR NBS 692

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	59.4800	59.4900	59.49750	.1225E-02
		2	59.5500	59.4800		
2	AU 2	1	59.6167	59.5171	59.53200	.3544E-02
		2	59.5171	59.4771		
3	AU 3	1	59.5557	59.5551	59.57323	.2034E-02
		2	59.6402	59.5419		
4	FR 1	1	59.6167	59.6167	59.59925	.1218E-02
		2	59.5469	59.6167		
5	FR 2	1	59.5500	59.6198	59.60235	.4466E-02
		2	59.6896	59.5500		
6	FR 3	1	59.6738	59.6322	59.61825	.1971E-02
		2	59.5939	59.5731		
7	FR 4	1	59.7296	59.5724	59.64025	.4382E-02
		2	59.6167	59.6423		
8	FR 5	1	59.6504	59.8097	59.75963	.2006E-01
		2	59.6409	59.9375		
9	GER 1	1	59.4900	59.5300	59.58500	.3717E-01
		2	59.4500	59.8700		
10	IT 1	1	59.7000	59.6500	59.75750	.1056E-01
		2	59.8800	59.8000		
11	IT 2	1	59.6800	59.5500	59.69750	.1216E-01
		2	59.8000	59.7600		
12	JP 1	1	59.6510	59.6920	59.66225	.2269E-03
		2	59.6660	59.6500		
13	JP 2	1	59.6440	59.5750	59.60950	.1587E-02
		2	59.5750	59.6440		
14	JP 2	1	59.6180	59.6330	59.61075	.7783E-03
		2	59.5700	59.6220		
15	JP 4	1	59.5470	59.6170	59.59600	.1111E-02
		2	59.6030	59.6170		
16	JP 5	1	59.7000	59.7200	59.67000	.5467E-02
		2	59.5600	59.7000		
17	JP 6	1	59.5640	59.5660	59.55100	.4120E-03
		2	59.5220	59.5520		
18	JP 7	1	59.5660	59.5670	59.56200	.4953E-03
		2	59.5310	59.5840		
19	US 1	1	59.4700	59.6000	59.52000	.5133E-02
		2	59.4500	59.5600		

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE# 4. LABRADOR NBS 692

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	50.757
CHI-SQUARE(.95,33)	47.407

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR BOTTLE VARIANCE

LAB- 9 BOTTLE- 2 C-CALC = .381 C-CRIT(2,34) = .267

LAB- 8 BOTTLE- 2 C-CALC = .307 C-CRIT(2,33) = .273

DOORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 1	ZERO WITHIN-BOTTLE VARIANCE
LAB- 4	ZERO WITHIN-BOTTLE VARIANCE
LAB- 8	COCHRAN'S TEST FOR BOTTLE VARIANCE
LAB- 9	COCHRAN'S TEST FOR BOTTLE VARIANCE

ISO/TC102/SC2/WG168 FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 4. LABRADOR NBS 692

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	14	.23247260E+00	.16605186E-01
BETWEEN BOTTLES	15	.76994970E-01	.51329980E-02
WITHIN BOTTLES	30	.85976008E-01	.28658669E-02
TOTAL	59	.39544358E+00	

F-CALC = 1.791 F-CRIT(.95;15,30) = 2.015

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 4. LABRADOR NBS 692

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	42.885
CHI-SQUARE(.95,18)	28.877

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 9 C-CALC = .326 C-CRIT(4,19) = .229

LAB- 8 C-CALC = .261 C-CRIT(4,18) = .239

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 8 COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB- 9 COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG168 FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 4. LABRADOR NBS 692

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	16	.28312166E+00	.17695104E-01
WITHIN LABS	51	.17030001E+00	.33392158E-02
TOTAL	67	.45342167E+00	

REPEATABILITY VARIANCE	.33392158E-02
REPEATABILITY STD. DEV.	.05778595
BETWEEN-LABORATORIES VARIANCE	.35889720E-02
BETWEEN-LABORATORIES STD. DEV.	.05990803
REPEATABILITY	.16353423
PERMISSIBLE TOLERANCE BETWEEN LABS	.20522047
GRAND MEAN	59.60584265
VARIANCE OF THE GRAND MEAN	.26022212E-03

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N559E
SAMPLE: 5. CANADA SCH-1

LABORATORY NO.	COUNTRY	BOTTLE NO.	ANALYTICAL RESULTS		LAB MEAN	LAB VARIANCE
1	AU 1	1	60.5100	60.4500	60.48750	.2025E-02
		2	60.5400	60.4500		
2	AU 2	1	60.6917	60.5940	60.60038	.4019E-02
		2	60.5637	60.5521		
3	AU 3	1	60.5619	60.6944	60.65830	.5896E-02
		2	60.6371	60.7398		
4	FR 1	1	60.6638	60.6638	60.63588	.1169E-02
		2	60.5940	60.6219		
5	FR 2	1	60.6670	60.5972	60.66700	.3248E-02
		2	60.7368	60.6670		
6	FR 3	1	60.6571	60.6079	60.70743	.1390E-01
		2	60.6874	60.8773		
7	FR 4	1	60.9021	60.6702	60.78250	.9191E-02
		2	60.7611	60.7966		
8	FR 5	1	60.5561	60.7246	60.69295	.9019E-02
		2	60.7156	60.7755		
9	GER 1	1	60.7600	60.9200	60.85000	.6800E-02
		2	60.8000	60.9200		
10	IT 1	1	60.7000	60.6000	60.72500	.1157E-01
		2	60.8600	60.7400		
11	IT 2	1	60.6800	60.6900	60.67000	.5133E-02
		2	60.5700	60.7400		
12	JP 1	1	60.6640	60.6680	60.69075	.1708E-02
		2	60.7520	60.6790		
13	JP 2	1	60.6500	60.6210	60.61100	.1734E-02
		2	60.5520	60.6210		
14	JP 3	1	60.6460	60.7050	60.68825	.1661E-02
		2	60.7370	60.6650		
15	JP 4	1	60.6640	60.6360	60.71275	.5885E-02
		2	60.7480	60.8030		
16	JP 5	1	60.8900	60.8900	60.87250	.2425E-02
		2	60.9100	60.8000		
17	JP 6	1	60.6570	60.6470	60.63100	.6153E-03
		2	60.6140	60.6060		
18	JP 7	1	60.6840	60.7260	60.70100	.1436E-02
		2	60.6560	60.7380		
19	US 1	1	60.3500	60.4200	60.41750	.2292E-02
		2	60.4600	60.4400		

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 5. CANADA SCH-1

BARTLETT'S TEST FOR HOMOGENEITY OF BOTTLE VARIANCE

CALCULATED STATISTIC	28.074
CHI-SQUARE(.95,33)	47.407

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

COORNBOS' TEST FOR BOTTLE MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR TWO-WAY ANOVA

LAB- 4	ZERO WITHIN-BOTTLE VARIANCE
LAB-16	ZERO WITHIN-BOTTLE VARIANCE

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 5. CANADA SCH-1

TWO-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	16	.64027501E+00	.40017188E-01
BETWEEN BOTTLES	17	.10062973E+00	.59193959E-02
WITHIN BOTTLES	34	.15775063E+00	.46397243E-02
TOTAL	67	.89865537E+00	

F-CALC = 1.276 F-CRIT(.95;17,34) = 1.933

TEST SAMPLE IS HOMOGENEOUS

ISO/TC102/SC2/WG15B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 5. CANADA SCH-1

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	16.870
CHI-SQUARE(.95,18)	28.877

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC102/SC2/WG16B FE-TOTAL TICL3 REDUCTION METHOD DOC. ISO/TC102/SC2 N569E
SAMPLE: 5. CANADA SCH-1

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	18	.81007674E+00	.45004263E-01
WITHIN LABS	57	.26916379E+00	.47221717E-02
TOTAL	75	.10792405E+01	

REPEATABILITY VARIANCE	.47221717E-02
REPEATABILITY STD. DEV.	.06871806
BETWEEN-LABORATORIES VARIANCE	.10070523E-01
BETWEEN-LABORATORIES STD. DEV.	.10035199
REPEATABILITY	.19447211
PERMISSIBLE TOLERANCE BETWEEN LABS	.31553686
GRAND MEAN	60.67377237
VARIANCE OF THE GRAND MEAN	.59216136E-03

APPENDIX 2

Abbreviations Used in this Report

σ_r = SIGMA-R - within-laboratory standard deviation.

σ_L = SIGMA-L - between-laboratories standard deviation

r* = R - repeatability

$= 2.83^{*} \sigma_r$

p** = permissible tolerance between laboratories

$= 2.83\sqrt{\sigma_L^2 + \sigma_r^2/2}$

COUNTRY ABBREVIATIONS

AU = Australia
CND = Canada
FR = France
GER = Germany
IND = India
IT = Italy
JP = Japan
SW = Sweden
USA = United States

* Repeatability, r, is the value below which the absolute difference between two independent single test (analysis) results produced by a laboratory on identical material using the same equipment within a short interval of time, using the standardized test method, may be expected to be with a 95% probability.

** Permissible tolerance between laboratories, P, is the value below which the absolute difference between two means of duplicate test results on identical material obtained by operators of different laboratories using the standardized test method, may be expected to lie with a 95% probability.